Application No. 10/572,994

DOCKET NO: ARZ-024630-US

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE OF APPLICATION OF:

NELSON, LLOYD A. ET AL.

SERIAL NO: 10/572 994

THIS APPLICATION IS A US NATIONAL

STAGE APPLICATION OF

PCT/US02/12961:

INTERNATIONAL FILING DATE: APRIL : PRIORITY DATE: APRIL 27, 2001

25, 2002

FOR: POLY BASIC ACID ESTERS AND THEIR USE IN FIBRE OPTIC CABLES

RENEWED PETITION UNDER 37 C.F.R. 1.47(a) FOR FILING WHEN INVENTORS

REFUSE TO SIGN OR CAN NOT BE FOUND

COMMISSIONER FOR PATENTS ALEXANDRIA, VIRGINIA 22313

SIR:

The DECISION ON PETITION UNDER 37 CFR 1.47(a) dated December 15, 2006 required that we submit: 1) payment of outstanding fees; and 2) supplemental materials to confirm that the inventor Nancy Mosby refuses to execute the declaration herein. Applicants have complied with the Examiner's requirements and request that the filing requirement be considered complete.

Applicants respectfully submit a Renewed Petition regarding inventor Nancy Mosby's refusal to join in the application. Further, Applicants submit the following documents as Exhibits in support of the Renewed Petition.

Exhibit A: A copy of the U S Search.com report for inventor Nancy Mosby.

Application No. 10/572,994

- Exhibit B: A copy of the April 27, 2007 letter from J. M. Gilbreth to Nancy Mosby.
- Exhibit C: A copy of the May 7, 2007 letter from J. M. Gilbreth to Nancy Mosby.
- Exhibit D: A copy of the Federal Express tracking confirmation for the April 27,
- 2007 letter from J. M. Gilbreth to Nancy Mosby.
- Exhibit E: A copy of the Federal Express tracking confirmation for the May 7, 2007
- letter from J. M. Gilbreth to Nancy Mosby.
 - Exhibit F: A copy of the May 8, 2007 letter from Nancy Mosby to J. M. Gilbreth.

REMARKS

In order to satisfy the Decision on Petition dated December 15, 2006, the Applicants must submit the following:

- (1) payment of the outstanding claims fees in the amount of \$1,410; and
- (2) a Renewed Petition containing supplemental materials to confirm that the inventor Nancy Mosby refuses to execute the declaration herein.

Regarding 1), Applicants authorize the Office to Charge the \$1410.00 in additional claim fees to Deposit Account No. 50-4222. Accordingly, Applicants respectfully submit that requirement 1) is now satisfied; and, respectfully request the Office to indicate the same.

Regarding 2), Applicants respectfully submit a Renewed Petition containing the supplemental material as requested by the Examiner. Accordingly, a Renewed Petition under 37 C.F.R. 1.47(a) follows.

REFUSE TO SIGN OR CAN NOT BE FOUND

Applicants previously submitted a Petition under 37 C.F.R. 1.47(a) for filing when inventors refuse to sign or can not be found on November 8, 2006 in the above-mentioned application. In response, a Decision on Petition under 37 C.F.R. 1.47(a) dated December 15, 2006 was sent to Applicants. The Examiner stated that before it can be concluded that the inventor refuses to execute the declaration herein, Applicants are required to send a copy of the complete application to the last known address of the inventor, accompanied by a request that the inventor either execute the declaration or expressly confirm her refusal to execute the declaration in this specific application. Applicants have completed the above requirements as requested and the facts surrounding the completion of said requirements are as follows:

- I) In an effort to find inventor Nancy Mosby's most recent address we retained an internet search company, US Search.com. On April 4, 2007, US Search.com sent us a report listing the current address for Nancy D. Mosby (see Exhibit A).
- II) On April 27, 2007 and again on May 7, 2007, I sent copies of the present application and a continuation application to Nancy Mosby at her current address via Federal Express, requesting that she either: 1) execute declarations for each of the above applications; or 2) send me an email or letter confirming her refusal to sign. A copy of the April 27, 2007 letter, Federal Express No. 860444162763 and a copy of the May 7, 2007 letter, Federal Express No. 860444162800 are attached as Exhibits B and C respectively.
- III) On April 30, 2007 Ms. Mosby received the April 27, 2007 letter (see Exhibit D).
- IV) On May 8, 2007 Ms. Mosby received the May 7, 2007 letter (see Exhibit E).

V) On May8, 2007 I received a letter from Ms. Mosby in response to my April 27, 2007 letter. In her letter, Ms. Mosby attached her April 23, 2002 letter (which was previously submitted in Applicant's November 8, 2006 Petition) and stated her "continued wishes to have nothing to do with the above referenced patent, Arizona Chemical, or anyone associated with Arizona Chemical." She further stated that "to me, the previous letter is crystal clear. I do not understand why this matter was not resolved years ago and consider it to be so now." (see Exhibit F).

VI) Executed declarations have not been obtained from inventor Nancy D. Mosby.

It is further requested that, in light of the above being responsive to Examiner's Decision on Petition dated December 15, 2006, the filing requirement be considered complete and the application proceed to examination.

Please charge the amount of \$1020.00 required for the request for extension of time to Deposit Account No. 50-4222. In the event any variance exists between the amount enclosed and the Patent Office charges for filing the above-noted documents, including any fees required under 37 C.F.R 1.136 for any necessary Extension of Time to make the filing of the attached documents timely, please charge or credit the difference to Deposit Account No. 50-4222. Further, if these papers are not considered timely filed, then a petition is hereby made under 37 C.F.R. 1.136 for the necessary extension of time.

If there are any questions concerning any of the above, please feel free to contact the undersigned.

Correspondence Client Number:	Respectfully Submitted			
(832) 485-3541 (phone) (713) 667-4424 (fax)	Registration 33,388 No. 15 - 2007			

Exhibit A

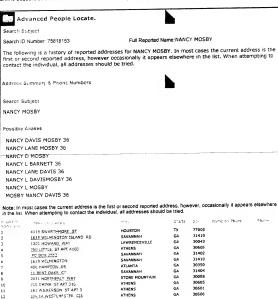
A copy of the U.S. Search.com report for inventor Nancy Mosby.

From: US Search Result < results@ussearch.com>

To: DEBBIE.SEGERS@COMCAST NET
Subject: Search Results from USSearch.com

Date: Wednesday, April 04, 2007 3:28:02 PM

Below are your search results. If you are having trouble viewing the report in this email, you may access it through your online account at www.ussearch.com/member or by clicking on the link provided in the confirmation email.



13 14	240 SEASRAVES DR 20 ORCHARD DR	ATHENS RINCON	GA GA	30605 31326	
Nation	al Death Record Search				
No Daco	rdr Tound				

US Search Customer Satisfaction Commitment: We hope you are satisfied with the results of your Advanced People Locate Search. We also realize that while these results will provide your search may require additional information. Because your satisfaction is important to us, US Search will extend a credit of \$30.00 lowards our Advanced Searches regarding.

*Credit expires in 30 days

\$30.00 Creuit towards one of the following services on "NANCY MOSBY

π	Name	Available Services (dick on a service below to learn more)	Purch	ase
1	NANCY MOSBY	Expert Assisted People Locate Search for "NANCY MOSBY". Location specialist performed search, Currier address and up to 10 year hastory and available isted ponce numbers. Relativus. Roundless and Negotics. Benicuptions: Tex feets, Small Claims Civil Juognents' Memage and Divortics', Real property ownership and value". Full name and possible alliess.	\$59.95 \$29.95	Buy
2	NANCY MOSBY	Expert Assisted Background Search for "NANCY MOSBY." Bishground Specialist performed search, Current address and up to 10 year instany. Available heled programmers. On Premise County Courthouse Search, Free Local and National media web-based search.	\$99.95 \$69.95	Buy

Other Popular Searches on NANCY MOSBY

#	Name	Available Services (click on a service below to learn more)	Purchase
1	NANCY MOSBY	Basic Background Search	Buy
2	NANCY MOSBY	Instant Criminal Records	

Exhibit B

A copy of the April 27, 2007 letter from J.M. Gilbreth to Nancy Mosby.

GILBRETH & ASSOCIATES, P.C.

J. M. (Mark) Gilbreth 124 Mary A. Gilbreth, Ph.D.⁴ Deborah G. Segers 134 Licensed to practice law in ¹TX, ²OK, and ³AR ⁴Registered, United States Patent & Trademark Office

April 27, 2007

Nancy D. Mosby 4115 Swarthmore Street Houston, Texas 77005 VIA FEDERAL EXPRESS NO. 8004 4416 2763

Re: ARZ - 024630-N1 (Application No. 11/637,206, PCT/US02/12961) Polybasic Acid Esters and Their Use in Cables

Dear Ms. Mosby.

Our firm represents Arizona Chemical Company in the above-referenced patent matter. Upon review of the files, it has come to our attention that you did not execute a declaration for these matters. We have enclosed a complete copy of the specification for the application listed above. Please sign the attached declaration, assignment, and power of attorney. Return the executed declarations to us in the Federal Express envelop we have provided for your convenience.

We noted from the files that you previously refused to execute the declarations. If you are still refusing to sign the declarations, please send me an email or sign this letter on the bottom and return in the FEDEX envelope, confirming your continuing refusal to sign the declarations for the above referenced matters.

By Friday, May 4, 2007 please either: (1) send the executed declarations to me or (2) send me an email or letter confirming your refusal to sign.

Thank you for your attention to this matter. If you need further assistance, you can call me at 713/667-1200 or email me at jmark@gilbreth.org.

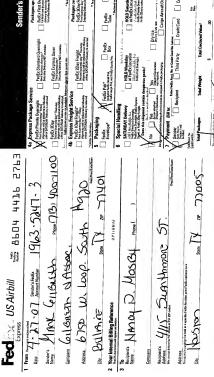
GILBRETH & ASSOCIATES, P.C.

April 27, 2007 Page 2

Very truly yours,

M. (Mark) Gilbreth

JMG/cc Enclosures



WHEREAS, We, LLOYD A. NELSON and NANCY D. MOSBY, are the inventors of POLY BASIC ACID ESTERS AND THEIR USE IN FIBRE OPTIC CABLES application, filed as

```
US Patent Application No. 60/286,880, filed 4/27/2001; PCT/USO2/12961, filed 4/25/2002; US Patent Application No. 10/572,994, filed 3/23/2006; and US Patent Application No. 11/637,206, filed 12/11/2006.
```

WHEREAS, ARIZONA CHEMICAL COMPANY, (herein after "ASSIGNEE"), is desirous of acquiring the entire right, title and interest in and to the aforesaid invention throughout the world, and all right, title and interest in, to and under any and all Letters Patent of the United States and all other countries throughout the world;

NOW, THEREFORE, for and in consideration of the sum of One Dollar (\$1.00) to us in hand paid by ASSIGNEE and for other good and valuable considerations, the receipt of which is hereby acknowledged, we, hereby sell, assign, transfer and set over to ASSIGNEE all right, title and interest in and to the said invention throughout the world, and said application for U.S. Letters Patent, and any and all divisions, continuations, continuations—in-part having subject matter related hereto, and reissues thereof, and any and all Letters Patent of the United States and foreign countries which may be granted therefor, the same to be held and enjoyed by ASSIGNEE for its own use and benefit, and for the use and benefit of its successors, assigns, or other legal representatives, to the end of the term or terms for which said Letters Patent of the United States or foreign countries are or may be granted or reissued, as fully and entirely as the same would have been held and enjoyed by me if this assignment and sale had not been made.

And we hereby authorize and request the Commissioner of Patents and Trademarks to issue any and all Letters Patent of the United States on said invention or resulting from said application and from any and all divisions, continuations, and reissues thereof, to ASSIGNEE as assignee of my entire interest, and hereby covenant that we have the full right to convey the entire interest herein assigned, and that we have not executed and will not execute any agreement in conflict herewith.

And we further hereby covenant and agree that we will, at any time, upon request, execute and deliver any and all papers that may be necessary or desirable to perfect the title of said invention and to such Letters Patent as may be granted therefor, to ASSIGNEE,

INITIALS LAN: _____

its successors, assigns, or other legal representatives and that if SASSIGNEE, its successors, assigns or other legal representatives shall desire to file any divisional or continuation applications or to secure a reissue of such Letters Patent, or to file a disclaimer relating thereto, will upon request, sign all papers, make all rightful oaths and do all lawful acts requisite for the filing of such divisional or continuation application, or such application for reissue and the procuring thereof, and for the filing of such disclaimer, without further compensation but at the expense of said assignee, its successors, or other legal representatives.

And we do further covenant and agree that we will, at any time upon request, communicate to ASSIGNEE, its successors, assigns or other legal representatives, such facts relating to said invention and Letters Patent or the file history thereof as may be known to us, and testify as to the same in any interference or other litigation when requested so to do, without further compensation but at the expense of said assignee, its successors, or other legal representatives.

EXECUTED THIS day of	, 2007
	LLOYD A NELSON
EXECUTED THIS day of	, 2007.
	NANCY D. MOSRY

Express Mail Label No. Page 1 of

Docket No.	
024630-N1	

Declaration and Power of Attorney For Patent Application

English Language Declaration

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

Polybasic Acid Esters and Their Use in Cables

application on which priority is claimed.

the specification of which

is attached hereto.

was filed on 12/11/2006

Application Number 11/637,206
and was amended on
(if applicable)
I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.
I acknowledge the duty to disclose to the United States Patent and Trademark Office all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, Section 1.56.
I hereby claim foreign priority benefits under Title 35. United States Code. Section 119(a)-(d) or

Section 365(b) of any foreign application(s) for patent or inventor's certificate, or Section 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate or PCT international application having a filing date before that of the

as United States Application No. or PCT International

1 1	nereby cla	aim 1	the	benefit	under	35	U.S.C.	Section	119(e)	of	any	United	States	provisional
	60	0/286,	880				4/2	7/2001						
	(Applica	ation	Seri	al No.)			(Fili	ng Date)						
	(Applica	ation :	Seri	al No.)			(Fili	ng Date)						
	(Applica	ation \$	Seria	al No.)			(Fili	ng Date)						

I hereby dalm the benefit under 35 U. S. C. Section 120 of any United States application(s), or Section 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. Section 112, I acknowledge the duty to disclose to the United States Patent and Trademark Office all information known to me to be material to patentability as defined in Title 37, C. F. R., Section 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application:

10/572,994	3/23/2006	
(Application Serial No.)	(Filing Date)	(Status) (patented, pending, abandoned)
(Application Serial No.)	(Filing Date)	(Status) (patented, pending, abandoned)
(Application Serial No.)	(Filing Date)	(Status) (patented, pending, abandoned)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

POWER OF ATTORNEY: As a named inventor. I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith. (Ilst name and registration number)

ATTORNEYS ASSOCIATED WITH CUSTOMER

NUMBER 50947

Send Correspondence to: ADDRESS ASSOCIATED WITH CUSTOMER NUMBER 50947

Direct Telephone Calls to: (name and telephone number)

MARK GILBRETH, 713 667 1200 X 102 OR MARYGILBRETH X 101

Full name of sole or first inventor LLOYD A. NELSON

Sole or first inventor's signature

Residence 107 TEAKWOOD DRIVE, SAVANNAH, GA 31410

Citizenship

USA

Post Office Address

same as residence

Full name of second inventor, if any NANCY D. MOSBY

Second inventor's signature

Residence

4115 SWARTHMORE STREET, HOUSTON, TX 77005

Citizenship USA

Post Office Address

same as residence

Date

024630-N1 Page 1 of 1

Continuation Application Checklist Form CHK011.0106 (rev. 0806) HTERNATIONAL (A) PAPER

CONTINUATION PATENT APPLICATION

(WITH CLAIM TO DOMESTIC PRIORITY)

INSTRUCTIONS: This form should be used for liting new continuation graterit applications. This form should not be used for provisional, priority, CIP, divisional or PC1 pattern applications not should him for used to fife an RCC. Check each how to indicate which terms on this checkstare included in the filting packet. Once the firm and increased in the control of the control of

is complete, print two (2) hardcopies, attach one (1) copy to the face of the application filing packet file copy and retain one (1) copy for expense tracking p	urposet	1
The following documents MUST be included with EACH new continuation patent application:		4
International Paper Cover Letter signed by: □ RCS □ DCS □ TW3 □ MME	12	
USPS Mail Label No. EQ952203670US		4
Form PTO SB/05 (Utility Patent Application Transmittal)	13	
Application Date Sheet, 2 sheet(s)	0	ý
Written Description (Background, Summary and Detailed Description): 19 sheet(s)	100	4
Electronic copy of Written Description saved on CORPLGL drive		
Patent Specification is a copy of the Patent Specification that was filed with the parent application		
Claims: independent claims + dependent claims, arranged on 4 sheets		
Patent Abstract: 1 sheet(s)		
Drawings: Figures arranged on sheet(s)	0	
OPTIONAL: Form PTO/SB/35 (Nonpublication Request)	0	
OPTIONAL/FILED IN PARENT: Form PTO/SB/22 (Extension of Time) - month(s), sheet(s)	C	
Authorization to charge Deposit Account in the amount of parent docket no.	-	-
Duplicate copy of Form PTO/SB/22 enclosed (when Deposit Account is charged)		
Return-receipt postcard	C	0
The following documents MAY be filed AFTER the initial filing:		
Form PTO/SB/17 (Fee Transmittal)	0	0
Authorization to charge Deposit Account in the amount of		
Form PTO/SB/08 (Information Disclosure Statement), sheet(s), including Copies of all references	0	50
English language references NOT copied in file. Foreign language references NOT copied in file.		
This IDS lists all references that were previously listed on an IDS submitted with the parent application.	170	
Signed Inventors Declaration: New, sheet(s) OR Copy from parent, sheet(s)	U	
(if a copy from the parent): Statement provided as to deletion of inventor(s) OR No statement requir	ed	
Preliminary Amendment, sheet(s)		
Claims (after amendment): independent claims + dependent claims. sheets		
Specification amended to include cross-reference to related application(s).		
Form prepared by Michele L. Wolfer on 12/11/2006 File Ref. ARZ-024630-N1		
Finin Foutin Finout Foutout		
Finin Foutin Finout Foutout Remarks/comments. 1041-06 Print two (2) copies of this completed form before closing this window. PRE-034630		
1 MS		
Print two (2) copies of this completed form before closing this window.		

Fried on 1 2006
Applicanits, loyd A Neison
Talle: Pely Basic And Esters and Their Use in Flore Opio: Céclies
Attorney Decker No. ARZ-024030-PM
USPN Express Mail Label (dated 1271) 2005
2U074E052P93
by the stamp piaced hereon, we acknowledge recogn of
Cover Letter,1 sheet(s). Unday Potent Applin Transmittal, Form PTOSB/05,1 sheet(s).
For Transmital, Form PTO/SB/17. sheet(s). Application Data Sheet, 2 nheet(s). Patent Specification, 24 sheet(s).
Figures arranged on shoot(s). Information Disclosure Statement, Form PTU/SB/08, shoot(s).
and one copy of each reference cited thereon.
a copy of the Inventors Declaration(s) filed in connection with the above-referenced Parent Application, shoot(s):
Preliminary Amendment, sheet(s)
Nonpublication Request, Form PTO/SB/35, sheetist
EXPRESS Customer Copy Label 11-B. March 2004
Post Office To Addressee





THOMAS W. BARNES, Ph.D. PATENT AGENT INTELLECTUAL PROPERTY CINCINNATI TECHNOLOGY CENTER 6285 TRI-RIDGE BOULEVARD LOVELAND OH 45140

T 513 248 6736 F 513 248 6455 thomas.barnes@ipaper.com

VIA USPS EXPRESS MAIL LABEL NO. EQ952203670US

11 December 2006

Commissioner for Patents Post Office Box 1450 Alexandria, Virginia 22313-1450

RE: SUBMISSION OF CONTINUATION PATENT APPLICATION

Applicant : Lloyd A. Nelson et al.

Title : Poly Basic Acid Esters and Their Use In Fibre Optic Cables

Our Ref. : ARZ-024630-N1

Continuation of USSN 10/572,994 filed on 03/23/2006 ("Parent Application")

Dear Commissioner

Enclosed herewith for filing, Applicant(s) respectfully submit(s) the following checked Items:

X	Utility Patent Application Transmittal, Form PTO/SB/05,1_ sheet(s);
	Fee Transmittal, Form PTO/SB/17, sheet(s);
\times	Fee Transmittal, Form PTO/SB/17, sheet(s); Application Data Sheet,3 sheet(s);
\boxtimes	Patent Specification, 24 sheet(s);
	Figures arranged on sheet(s);
	Information Disclosure Statement, Form PTO/SB/08, sheet(s), and one copy
	of each reference cited thereon;
	a copy of the Inventors Declaration(s) filed in connection with the above-referenced
	Parent Application, sheet(s);
	Preliminary Amendment, sheet(s);
7	Nonpublication Request, Form PTO/SB/35, sheet(s);
7	Other: ; and,
<u> </u>	a postage-prepaid, return-receipt postcard with a listing of the above-checked items

Please stamp the enclosed postcard and return same to me to indicate your receipt of the above-listed items. Please feel free to contact me if you have any questions concerning the above or the enclosed.

With kindest regards.

Thomas W. Barries
Agent for Applicant(s)

Enclosures TWB/mlm

PTO/SB/05 (04-05)

Approved for use through 97/31/2005 OMB 0951-0532
U.S. Pateril and Trademark Office U.S. DEPARTMENT OF COMMERCE

Under the Paperwork Reduction Act of 1995, no persons are required to r	respond to a collection of inform	
UTILITY	Attorney Docket No	ARZ-024630-N*
PATENT APPLICATION	First Inventor	Lloyd A. Nelson
TRANSMITTAL	Title	Poly Basic Acid Esters and Their
(Only for new nonprovisional applications under 37 CFR 1.53(b))	Express Mail Label No	EQ852203670US
APPLICATION ELEMENTS See IMEE crapter 600 concerning utility patent application contents	ADDRESS TO:	Commissioner for Patents P.O. Box 1450 Alexandria VA 22313-1450
1. Fee Transmittal Form (e.g., PTO/SB/17)	ACCOMPAN	IYING APPLICATION PARTS
(Submit an original and a duplicate for fee processing) Applicant claims small entity status.	Assignment Papers (cover sheet & document(s)) Name of Assignee	
See 37 CFR 1.27. 3 Specification [Total Pages 24]		
Both the claims and abstract must start on a new page (For information on the preferred arrangement, see MPEP 605 01(a)) Drawling(\$1 (35 U.S C 113) Total Sheets	Traine of risa	9111.1
5. Oath or Declaration [Total Sheets]	10 37 CFR 3.73(b)) Statement Power of san assignee) Attorney
A copy from a prior application (37 CFR 1.63(d)) for continuation/divisional with Box 18 completed)	11 English Transi	lation Document (if applicable)
DELETION OF INVENTOR(S) Signed statement attached deleting inventor(s)	12. Information Di	sclosure Statement (PTD/SB/08 or PTD-1449
name in the prior application, see 37 CFR 1 63(d)(2) and 1.33(b).	Copies	of citations attached
6 Application Data Sheet. See 37 CFR 1.76	13 Preliminary An	nendment
CD-ROM or CD-R in duplicate, large table or Computer Program (Appendix)	14. Return Receipt Postcard (MPEP 503)	
Landscape Table on CD	(Should be sp	ecifically itemized)
Nucleotide and/or Amino Acid Sequence Submission (if spaicable, ilems a. – c. are required)	15. Certified Copy (if foreign prior	of Priority Document(s) rity is claimed)
a Computer Readable Form (CRF) b. Specification Sequence Listing on	16. Nonpublication	Request under 35 U.S.C 122(b)(2)(B)(i).
CD-ROM or CD-R (2 copies); or	Applicant mus	t attach form PTO/SB/35 or equivalent.
ii Paper	17. C Other: cover lett	er
c Statements verifying identity of above copies 18. If a CONTINUING APPLICATION, check appropriate box, and supp	bly the requisite information	below and in the first sentence of the
specification following the title, or in an Application Data Sheet under 37	CFR 1.76	
		or application No. 10/572,994
Prior application information: Examiner	Art Unit	·
19. CORRESPOND	ENGE ADDRESS	
The address associated with Customer Number. 0172	e6 o	R Correspondence address below
Name		
Address		
City Stale		Zip Code
Country Telephone		Email
Signature / // . 53555	Date	Desired to be
Name (Print/Type) Therries W. Barnes III, Ph.D.		Registration No (Attorney/Agent) 52.595

This oblican of information is required by T. CER. 15.5(b). The information is required to dature or return a world by the public which is to fix and by the USETO its more of an approximation. Ordinatelysis is governed by \$0.1.5.C. 12 and \$0.7 RR. 11 and \$1.7 R. collection is externed to law 17 minutes to complete, including gathering, preparing, and superhing the completed application form to the USPTO. Time will vary oppositing upon the industrial cases. Any comments to the second of one poly readings to complete the form model or supportions or industrial, including cases in the ordination of the USPTO. The ordination is second to the USPTO. The ordination is second to the ordination of the USPTO. The ordination is second to the ordination of the USPTO. The

Application Data Sheet

Application Information

Application Type:: Regular Subject Matter:: Utility

Title:: POLY BASIC ACID ESTERS AND

THEIR USE IN FIBRE OPTIC

CABLES

Attorney Docket Number:: ARZ-024630-N1

Request for Early Publication?:: No
Request for Non-Publication?:: No
Total Drawing Sheets:: 0
Small Entity:: No

Applicant Information

Applicant Authority Type:: Inventor

Primary Citizenship Country:: US

Status:: Full Capacity

Given Name:: Lloyd
Middle Name:: A
Family Name:: Nelson
City of Residence:: Savannah
State/Province of Residence:: Georgia
Country of Residence:: US

Street of Mailing Address:: 107 Teakwood Drive

City of Mailing Address:: Savannah
State/Frovince of Mailing Address:: Georgia
Country of Mailing Address:: US
Postal/Zip Code of Mailing Address:: 31410

Applicant Information

Applicant Authority Type:: Inventor Primary Citizenship Country:: US

Status:: Full Capacity

Giver Name:: Nancy
Middle Name:: D

Family Name:: Mosby
City of Residence:: Savannah
State/Province of Residence:: Georgia

Country of Residence:: US

Street of Mailing Address:: 1619 Wilmington Island Road

City of Mailing Address:: Savannah
State/Province of Mailing Address:: Georgia
Country of Mailing Address:: US
Postal/Zip Code of Mailing Address:: 31410

Correspondence Information

Correspondence Customer Number:: 01726

Phone Number:: 513-248-6736 Fax Number:: 513-248-6455

E-Mail Address:: Thomas.barnes@ipaper.com

Representative Information

Representative Customer Number:: 01726

Domestic Priority Information

Application::	Continuity	Parent	Parent Filing
	Type::	Application::	Date::
This application is	Continuation of	10/572,994	03/23/2006
10/572,994	National Stage of	PCT/US02/12961	4/25/2002
PCT/US02/12961	An application claiming the benefit under 35 USC 119(e)	60/286,880	4/27/2001

POLYBASIC ACID ESTERS AND THEIR USE IN CABLES

BACKGROUND OF THE INVENTION

Field of the Invention

This invention generally relates to protecting moisture-sensitive substrates from moisture, to articles such as a fiber optic cable that contain both a moisture sensitive component and a protectant composition; and to compositions that may be used to protect substrates from moisture and/or corrosion.

Description of the Related Art

as telecommunication devices (e.g., service or power wires/cables) as well as telecommunication devices (e.g., fiber optic cables, telephone cables, terminal blocks, junctions and connections) are often exposed to harsh environmental conditions. Yet, both transmission cables and telecommunication devices are required to maintain uninterrupted mechanical and electrical characteristics despite the surrounding environmental conditions. Historically, transmission cables have been strung above ground between poles. Increasingly, these cables are being buried beneath the ground for aesthetic reasons and to protect them from extremes of temperature, rain, snow, ice, high whids, falling tree limbs and the like. However, even when placed underground, these cables are subject to many environmental stresses including mechanical shock during back filling, moisture, the possibility of water immersion, attack from rodents and exposure to salt and other corrosive materials.

Transmission cables typically contain either a bundle of individually insulated opper wires, or a bundle of fiber optic cables. The copper wires, in particular, are often protected by an inner metal shield, which covers the bundle of wires, and an outer sheath made from plastic or other insulating material. Likewise, a communication cable is generally constructed of an outer plastic jacket and an inner metal core wrap with an annular space there between. See, e.g., U.S. Patent No.3,745,321 to Eager, Jr. et al. The core wrap enwraps a plurality of twisted insulated conductors with a filler material, such as petroleum, wax, or other hydrocarbons, located between the conductors. The filler material byciscally functions as an insulator.

Even though wires and cables are normally well protected, if the protective outer sheath is cut or otherwise broken, water can seep into the wire or cable. Whenever moisture is present in the interior of a cable or wire, it tends, over a period of time, to migrate or flow longitudinally into connections at the splice closures, terminals, or the like. In the special case of optical fibers, passage of the water to connection points or terminals and associated equipment will typically result in damage to such equipment, especially to any metal parts thereof, and can also cause problems at low temperature or freezing environments due to fiber microbending. This is particularly true when an immersed wive is cut.

In an inadequately protected device, corrosion of the contacts or short circuits caused by moisture or other outside elements will eventually interrupt service. Replacement or repair of the cables, whether strung above the ground or buried, can be difficult and/or expensive, and the interruption of service may be particularly irritating to the customer. Because of this, much attention has focused on the protection of these cables from both physical damage and from chemical attack. As a result, various techniques have been disclosed to prevent or restrict migration of moisture along the internal passages of a cable.

20 pressurized gas. Upon escape of gas through a ruptured cable jacket, an alarm provides notice of cable damage. However, the gas does not prevent the flow of water into and along the cable internal passages before the repair is effected.

U.S. Patent Nos. 4,867,526; 5,082,719; and 5,163,115 disclose a super-absorbent polymer or tape which is impregnated with a superabsorbent material for preventing water migration through cables. It has been found that, for a number of reasons, the production and/or installation of these materials is often impractical and/or uneconomical.

Silicone grease lubricant, elastomeric seals, and/or mastic sealing strips have been used in an attempt to seal out moisture. While these sealants offer some protection, moisture can often seep into the enclosure and cause a short circuit, which will interrupt telecommunications service, particularly when the terminal block is buried underground.

It is also known that insulating materials, such as mineral oil, protect the conductor from moisture and corrosive materials. However, these such insulating materials drip when the cable is being serviced (i.e., cut). To evercome this "dripping" problem, various pelling agents are known to be added

PCT/US02/12961 WO 02/088208

to the insulator, to thereby provide a gelled, non-dripping protectant composition. See, e.g., U.S. Patent Nos. 6,169,160; 6,160,939; and 6,085,009. Zeolites have recently been suggested as a suitable material to

impart moisture-resistance to a cable. See. e.g., US 6,205.276 B1.

Hydrophobic furned silica has been used as a gelling agent, particularly for fiber optic cables. See, e.g., U.S. Patent Nos. 5,905,833; 5.902.849; 5.737.469; 5.285,513; 5,276,757; and 5,187,763; and European Patent FP 0 206 234 B1. The furned silica, in combination with a suitable fluid, will cause the fluid to become gelled and/or demonstrate thixotropic properties. 10 which is advantageous for a cable protectant composition. Furned silica is rather expensive, and so suitable fluids that are relatively less expensive have a commercial advantage.

Due to the relatively long lengths of cable that are typically installed, certain cable components are made from plastic components such as 15 oplypropylene in an attempt to both reduce the cost of the cable as well as obtain multiple supply sources for such components. This presents a problem to the supplier of protectant compositions in that many known protectant compositions are not compatible with plastics such as polypropylene and can cause deterioration of those materials. Furthermore, although only a fairly small amount 20 of protectant composition is present in a cross-section of cable, because the cables are extremely long, even a small difference in the per pound cost of the protectant composition can have a large commercial advantage in the marketplace, assuming all other performance properties are met.

A need therefore exists for suitable and cost-effective protectant 25 compositions to be used in transmission cables and telecommunication devices. The present invention fulfills these needs and further provides related advantages.

SUMMARY OF THE INVENTION

5

In one aspect, the present invention provides a composition 30 comprising a blend of first and second esters: the first ester comprises a reaction product of first reactants; the first reactants comprise trimer acid, first polyhydric alcohol and first monohydric alcohol; the first monohydric alcohol is selected from the group of C8-18 monohydric alcohols; and the second ester comprises a reaction product of second reactants; the second reactants 35 comprise trimer acid, second polyhydric alcohol and second monohydric

PCT/US02/12961 WO 02/088208

alcohol; the second monohydric alcohol is selected from the group of C₆₋₁₀ monohydric alcohols. The first and second monohydric alcohols are nonidentical.

In another aspect, the present invention provides a method for 5 preparing an ester composition having a viscosity in the range of 30-50 cSt (centistokes) at 100°C. The method comprises a) preparing a first ester, the first ester comprising a reaction product of first reactants, the first reactants comprising trimer acid, first polyhydric alcohol and first monohydric alcohol. where the first monohydric alcohol is selected from the group of Calar 10 monohydric alcohols; b) preparing a second ester, the second ester comprising a reaction product of second reactants, the second reactants comprising trimer acid, second polyhydric alcohol and second monohydric alcohol, where the second monohydric alcohol is selected from the group of C₆₋₁₀ monohydric alcohols; and c) blending the first and second esters together in a proportion to 15 provide an ester composition having a viscosity in the range of 30-50 cSt at 100°C; with the proviso that the first and second monohydric alcohols are nonidentical.

The present invention also provides a composition prepared by the above-described method.

In another aspect, the present invention provides a cable-filling composition comprising silica and a blend of first and second esters. The first ester comprises a reaction product of first reactants, the first reactants comprising trimer acid, first polyhydric alcohol and first monohydric alcohol, where the first monohydric alcohol is selected from the group of C8-18 25 monohydric alcohols. The second ester comprises a reaction product of second reactants, the second reactants comprising trimer acid, second polyhydric alcohol and second monohydric alcohol, where the second monohydric alcohol is selected from the group of C₆₋₁₀ monohydric alcohols. The first and second monohydric alcohols are non-identical.

20

30

In another aspect, the present invention provides a method for insulating the contents of a cable, e.g., a fiber optic cable. The method comprises: a) preparing a cable-filling composition comprising silica and a blend of first and second esters; the first ester comprising a reaction product of first reactants, the first reactants comprising trimer acid, first polyhydric alcohol 35 and first monohydric alcohol, where the first monohydric alcohol is selected from the group of C₈₋₁₈ monohydric alcohols; the second ester comprising a

reaction product of second reactants, the second reactants comprising trimer acid, second polyhydric alcohol and second monohydric alcohol, where the second monohydric alcohol is selected from the group of C₆₋₁₀ monohydric alcohols; the first and second monohydric alcohols being non-identical; and b) placing the cable-filling composition of step a) into a cable, e.g., a fiber optic cable.

The present invention also provides a fiber optic cable comprising a cable-filling composition; the cable-filling composition comprising silica and a blend of first and second esters; the first ester comprising a reaction product of first reactants comprising trimer acid, first polyhydric alcohol and first monohydric alcohol; the first monohydric alcohol selected from the group of C₉₋₁₉ monohydric alcohol; the second ester comprising a reaction product of second reactants comprising trimer acid, second polyhydric alcohol and second monohydric alcohol; the second monohydric alcohol selected from the group of C₅₋₁₀ monohydric alcohols; wherein the first and second monohydric alcohols are non-identical.

These and other aspects of the present invention are described in further detail below.

DETAILED DESCRIPTION OF THE INVENTION

20 In one aspect, the present invention provides a composition comprising a blend of first and second esters, wherein the first ester comprises a reaction product of first reactants comprising trimer acid or a reactive equivalent thereof, first polyhydric alcohol or reactive equivalent thereof and first monohydric alcohol being selected 25 from the group of C₈₋₁₆ monohydric alcohols or reactive equivalent thereof; the second ester comprises a reaction product of second reactants comprising trimer acid or reactive equivalent thereof, second polyhydric alcohol or reactive equivalent thereof in the second monohydric alcohol or reactive equivalent thereof; the second monohydric alcohol or reactive equivalent thereof; the second monohydric alcohols. The first and second monohydric alcohols are non-identical. Before further describing these compositions of the present invention, the reactants used to prepare the components thereof will be discussed.

Unsaturated fatty acids, as obtained from, e.g., wegetable oils, tallow, and tall oil (the latter being known as tall oil fatty acids, or TOFA) may be subjected to thermal polymerization typically in the presence of a clay catalyst.

PCT/US02/12961 Vi O 02/088208

to provide a product known commercially as polymerized fatty acid. To a large extent, the polymerized fatty acid is the dimerization and trimerization product of the fatty acid. Because these fatty acids typically contain 18 carbons, the corresponding dimerized polymerized fatty acids, also called dimer acid, 5 contains 36 carbon atoms. The corresponding trimerized polymerized fatty acids, also called trimer acid, contains 54 carbon atoms. The dimer and trimer acids are a mixture of complicated isomeric structures. Detailed descriptions of polymerized fatty acid, in terms of product, composition and uses, may be found in, for example, Naval Stores - Production, Chemistry and Utilization, D.F. 10 Zinkel and J. Russel (eds.), Pulp. Chem. Assoc, Inc., 1989, Chapter 23 and the Encyclopedia of Chemical Technology II, Vol. 8, pages 847-848. See also, U.S. Patent No.s 2,904,415; and 2,482,761, and Industrial and Engineering Chemistry, 32:802 (1940).

The polymerization of fatty acids typically provides a mixture of 15 dimer acid and trimer acid. These two materials have different boiling points and can be separated from one another by distillation. For instance, dimer can be distilled away from other polymerized fatty acids at a temperature ranging from 250-280°C (at 0.3 to 0.5 mmHg) while trimers may be distilled from other polymerized fatty acids at a temperature ranging from 280-310°C (at 0.005 to 20 0.01 mmHg). Accordingly, by suitable distillation a mixture of polymerized fatty acids can provide trimer acid in various purities, also known as grades. For the present specification, the term "trimer acid" will be used to designate a composition containing polymerized carboxylic acid which is at least 20% trimer based on the total weight of the polymerized fatty acid in the 25 composition. Typically, it is difficult to refine trimer acid to a purity of greater than 80%. Accordingly, a preferred trimer acid of the present invention is contained within a mixture of polymerized fatty acids, where the polymerized fatty acids also includes dimer acid, and the weight ratio of dimer acid: trimer acid ranges from 20:80 to 80:20.

A reactive equivalent of trimer acid is a material that may be used in place of trimer acid to provide essentially the same ester product in the esterification reaction of the present invention. Hydrogenated trimer acid is a reactive equivalent of trimer acid. As initially formed, dimer acid and trimer acid typically contain unsaturated polybasic acids. This unsaturation may, to some 35 degree he eliminated by subjecting the dimer acid/trimer acid to hydrogenation under suitable reaction conditions whereby the unsaturation reacts with

30

hydrogen and becomes saturated. Trimer acid that has been subjected to a hydrogenation process is commonly referred to as hydrogenated trimer acid, and is well known in the naval stores industry. Hydrogenated trimer acid is typically available from the same commercial suppliers who sell trimer acid and dimer acid. Hydrogenated trimer acid typically has a lighter color than "standard" non-hydrogenated trimer acid, and thus its use in an esterification process provides lighter colored esters. In addition, esters prepared from hydrogenated trimer acid typically display enhanced thermal stability. Accordingly, hydrogenated trimer acid is a preferred trimer acid for use in the 10 present invention.

Another reactive equivalent of trimer acid is a partial or complete esterification product of trimer acid. For example, trimethyl trimerate and triethyl trimerate are esterification products of trimer acid that may be used in the present invention. When employed in the present invention, a trimerate ester of trimer acid will effectively react to provide the same product as is obtained from trimer acid itself. The esterification product will, however, generate methanol or ethanol, or whatever other alcohol was used to esterify the trimer acid. This alcohol is preferably of low molecular weight and/or high volatility so that it can be readily distilled from the reaction mixture during preparation of the composition of the present invention. Another reactive equivalent is the acid halide of trimer acid.

Many companies currently manufacture and sell polymerized fatty acids that include trimer acid according to the present invention. For example, Arizona Chemical (Jacksonville, Ft.; @artzonachemical.com) sells UNIDYME® 40 dimer acid, Jacksonville, Ft.; @artzonachemical.com) sells UNIDYME® 60 trimer acid abt contains about 67% trimer acid and 33% dimer acid. Additional current suppliers of trimer acid and trimer acid-containing polymerized fatty acids include Henkel Corporation, Emery Oleochemicals Division (Cincinnati, OH, with their EMPOL™ line of dimer and trimer acids) and Uniquema North America (Wilmington, DE, with their PRIPOL™ line of dimer and trimer acids).

In one aspect, each of the first reactants and the second reactants further comprise dimer acid. For example, the first reactants and the second reactants each may comprise dimer acid and trimer acid, in a dimer acid:trimer acid weight ratio of 20:80 to 80:20, the weight ratio independently selected in each of the first and second reactants. In one aspect, at least one of the reactants

employs a blend of dimer acid and trimer acid wherein the trimer acid is present at a greater weight percent, i.e., greater than 50 wt%, than the dimer acid.

The polyhydric alcohol, which may also be referred to as a polyol, has two or more hydroxyl groups. Thus, the polyol has of the chemical formula 5 R¹(Ofh), wherein R¹ is an n-valent organic group. Optionally, R¹ is a C₂-C₂e organic group without hydroxyl substitution, and preferably R¹ is a C₂-C₂e organic group. Suitable polyhydric alcohols include, without limitation, ethylene glycol, propylene glycol, neopentyl glycol, butyleneglycol, glycerol, trimethylolpropane, pentaerythritol, tris(hydroxylmethylmethylol), dipentaerythritol, and tri-pentaerythritol. In one aspect of the invention, the first and second polyhydric alcohols are each independently selected from pentaerythritol, di-pentaerythritol, tri-pentaerythritol, trimethylolpropane, ethylene glycol and neopentyl glycol. In one aspect, at least one of the first and second polyhydric alcohols is neopentyl glycol (CAS No. 126-30-7), also known as 2,2-dimethyl-1,3-propanediol, while in a preferred embodiment the first and second polyhydric alcohols are each neopentyl glycol. Again, a reactive equivalent could be used, e.g., the acetate ester of the polyol.

The monohydric alcohol has a single hydroxyl group. Thus, the monohydric alcohol may be represented by the chemical formula R²-OH where 20 R² is an organic group containing carbons. The identity of the monoalcohol has a very important impact on the viscosity of the ester produced from the monoalcohol in combination with the polyol and trimer acid. In general, as the molecular weight of the monoalcohol increases, the viscosity of the ester derived therefrom increases.

25 The present invention provides a blend of first and second esters. The first ester is prepared from a first monoalcohol while the second ester is prepared from a second monoalcohol.

The first monoalcohol is a C₈₋₁₆ monohydric alcohol. In other words, the first alcohol is an organic compound having at least 8, and no more 30 than 18, carbon atoms, and a single hydroxyl (OH) group. The first monoalcohol may be represented by the formula R²-OH, where R² is a hydrocarbon having 8-18 carbons. Suitable examples include 1-octanol, 1-decanol, 1-dedeanol (a.k.a. lauryl alcohol), 1-tridecylalcohol, 1-tetradecanol (a.k.a. myristyl alcohol), palmitlyl alcohol, and stearyl alcohol. The first alcohol is may be linear or branched. In one aspect of the invention, the first alcohol is a branched alcohol. A preferred branched alcohol is iso-tridecyl alcohol, available

from ExxonMobil Chemicals as EXXAL™ 13 alcohol. In another aspect, the first monohydric alcohol is selected from C_{10:14} primary monohydric alcohols.

The second monoalcohol is a C₆₋₁₀ monohydric alcohol. In other words, the second alcohol is an organic compound having at least 6, and no more than 10, carbon atoms, and a single hydroxyl (OH) group. The second monoalcohol may be represented by the formula R³-OH, where R³ is a hydrocarbon having 6-10 carbons. The second alcohol may be linear, branched, or cyclic. Suitable examples include 1-hexanol, 1-heptanol, 1-octanol, 1-nonanol, 1-decanol, 2-hexanol, 2-heptanol, 2-betanol, 2-heptanol, 2-nonanol, 2-hexanol, 2-heptanol, and cyclocatanol. In one aspect of the invention, the first alcohol is a branched alcohol. A preferred branched alcohol is 2-ethylhexanol. In another aspect, the second monohydric alcohol is selected from 2-ethylhexanol, 2-octanol and cyclohexyl alcohol.

Both monohydric and polyhydric alcohols useful in the present in invention are very well known in the art and may be obtained from many commercial suppliers including, for example, Aldrich (Milwaukee, WI; @aldrich.sial.com); EM Industries, Inc. (Hawthore, NY; @emscience.com); Lancaster Synthesis, Inc. (Windham, NH; @lancaster.co.uk); and Spectrum Quality Product, Inc. (New Brunswick, NJ; @spectrumchemical.com).

20 The first ester comprises the reaction product of reactants comprising timer acid, first polyhydric alcohol and first monohydric alcohol or reactive equivalents of one or more. This first ester has a low acid number, that is, an acid number of less than 25, preferably less than 20, 15, 10, 5, 2, or 1. It has a viscosity, as measured in centistokes (cSt) that varies depending on the temperature of measurement. At 40°C, the first ester preferably has a viscosity of about 410-510 cSt, more preferably about 430-490 cSt, and still more preferably about 430 cSt. When measured at 100°C, the first ester preferably has a viscosity of about 35-45 cSt, more preferably about 37-43, and still more preferably about 40 cSt. The viscosity index (VI) of the first ester is preferably about 120-150, more preferably about 130-140, and still more preferably about 120-150, more preferably about 130-140, and still more preferably about 130-140.

The second ester comprises the reaction product of reactants comprising trimer acid, second polyhydric alcohol and second monohydric alcohol or reactive equivalents of one or more. This second ester has a low acid number, that is, an acid number of less than 25, preferably less than 20, 15, 10, 5, 2, or 1. It has a viscosity, as measured in certifictokes (cSt) that varies

PCT/US02/12961 WO 02/088208

depending on the temperature of measurement. However, at any temperature below 100°C, the first ester preferably has a greater viscosity than the second ester. At 40°C, the second ester preferably has a viscosity of about 280-380 cSt, more preferably about 300-360 cSt, and still more preferably about 330 cSt. 5 When measured at 100°C, the second ester preferably has a viscosity of about 28-38 cSt, more preferably about 30-36, and still more preferably about 33 cSt. The viscosity index (VI) of the second ester is preferably about 125-160, more preferably about 130-150, and still more preferably about 145.

In a preferred aspect of the present invention, the first monohydric 10 alcohol is selected from the group of C₁₀₋₁₅ primary monohydric alcohols; the second monohydric alcohol is selected from the group consisting of Ca monohydric alcohols; and the first and second polyhydric alcohols are selected from C₃₋₁₅ polyhydric alcohols.

In one aspect, the present invention provides a blend of two esters. 15 namely first and second esters, wherein the first ester has a greater viscosity than the second ester. In one aspect, the blend has a viscosity of 30-50 cSt at 100°C, while in another aspect the blend has a viscosity of about 40 cSt at 100°C. In a preferred embodiment, the first monohydric alcohol is selected from the group of C₁₀₋₁₅ primary monohydric alcohols; the second monohydric alcohol 20 is selected from the group consisting of C₈ monohydric alcohols; the first and second polyhydric alcohols are selected from C₃₋₁₅ polyhydric alcohols, and the viscosity of the composition is in the range of 30-50 cSt at 100°C.

The blend of two esters according to the present invention preferably has a low acid number, that is, an acid number of less than 25. 25 preferably less than 20, 15, 10, 5, 2, or 1. The blend has a viscosity, when measured at 100°C, of 34-46 cSt, preferably about 36-44 cSt, and more preferably about 40 cSt. The blend is readily prepared from component esters by mixing the esters together in single vessel, preferably with some stirring to expeditiously achieve a homogeneous blend.

An ester of the present invention is readily prepared by combining the reactants, namely trimer acid, polyhydric alcohol and monohydric alcohol or a reactive equivalent of one or more, and heating this mixture until water is evolved. Any order of combination is suitable, and heating rate is not particularly important. The final heating temperature is suitably about 200°C to 35 about 250°C. The progress of the reaction may be monitored by taking

30

samples and measuring the acid number and molten viscosity of those samples.

Upon heating, water vapor will typically be evolved as the esterification reaction occurs. Preferably, the water vapor is condensed and 5 removed from the reaction mixture as soon as it forms, thus driving the reaction to completion. A Dean-Stark trap is suitably used for this purpose. Alternatively, the water vapor is removed by application of a modest vacuum, e.g., about 20-200 mm.

A catalyst may be used to speed up the rate of the esterification 10 reaction, where suitable catalysts are well known in the art and include sulfuncacid, phosphoric acid and other inorganic acids, metal hydroxides and alkoxides such as tin oxide and titanium isopropoxide, and divalent metal salts such as tin or zinc salts. A preferred catalyst is a tin catalyst, e.g., FASTCAT™ 2001 catalyst. When a catalyst is present, it should be used in small amounts, e.g., 15 less than about 5 weight percent of the total mass of the reaction mixture. preferably less than about 2% and more preferably less than about 1% of the total mass of the reaction mixture. Excessive amounts of catalyst increase the cost of preparing the ester, as well as often leave behind residue that may be harmful to the environment in which the ester is located, e.g., within a cable.

In preparing a preferred cable-protectant composition of the present invention, the blend of first and second esters as described above is combined with furned silica. The cable-protectant composition provided herein preferably includes a high purity silicon dioxide which may be selected from amorphous silica, crystalline silica, fumed silica, flint, quartz, and combinations 25 thereof. A preferred silicon dioxide is a fumed silica.

20

35

Suitable furned silica is commercially available from a number of sources, including Cabot Corporation (Cabot Corporation, Boston, MA; @ cabot-corp.com: which sells CAB-O-SIL™ fumed silicas). Wacker Silicons Corp (Adrian, MI; @.wacher.com, a division of Wacker-Chemie.) Tulco Inc. 30 (Aver. MA; @.tulcocorp.com), and Degussa-Hüls AG (Frankfurt am Main, Germany; @.degussa-huels.de; which sells AEROSIL™ furned silica). Furned silica is also described in U.S. Patent Nos. 6.193.944; 5.910.295; and 5,902,636. Furned silica may be prepared by the high-temperature oxidation of a halosilane such as tetrachlorosilane, trichlorosilane, or methyltrichlorosilane. An antioxidant is an optional component that may be combined

with the ester blend and silica to prepare a cable-protectant composition of the

N O 02/088208 PCT/US02 12961

present invention. If present, the antioxidant is used in a small but effective amount which is generally on the order of up to about 2 parts by weight based on the entire weight of the composition. Examples of suitable antioxidants include phenolic-based and phosphite-based antioxidants. Suitable 5 antioxidants are well known in the art, where specific examples include, without limitation, IRGANOX™ 1010, IRGANOX™ 1076, and IRGANOX™ 1035 antioxidants (each from Ciba Specialty Chemicals, Tarrytown, NY or Basel, Switzerland; @cibasc.com) and MARK™ 2112, MARK™ 1500 and MARK™ 5014 antioxidants (each from Witco Corporation, Greenwich, CT; @witco.com)

The furned silica, in combination with a blend of esters according to the present invention, will form a gelled mixture and/or demonstrate thisotropic properties, which is advantageous for a cable protectant composition. Methods to combine furned silica with fluids other than the estercontaining compositions of the present invention, to form a cable protectant 15 composition, are well known in the art, and are equally applicable to the formation of cable protectant composition using the ester-containing compositions of the present invention. See U.S. Patent Nos. 5,905,833; 5,902,849; 5,737,469; 5,285,513; 5,276,757; and 5,187,763; and European Patent EP 0 206 234 B1, and references cited therein, for descriptions of these 20 methods

10

The following examples are provided for purposes of illustration and are not limiting on the present invention. In the Examples, UNIDYME™ 40 and 60 polymerized fatty acids are blends of dimer and trimer acid available from Arizona Chemical (Jacksonville, FL; @arizonachemical.com). 25 LINIDYME™ 40 polymerized fatty acid is a distillation product from the polymerization of unsaturated tall oil fatty acids, in the form of a dark viscous liquid (Gardner color 12, viscosity at 210°F of 140 cSt), having a 35% contents of trimer acid (by gas chromatography) and a 65% contents of dimer acid. UNIDYME™ 60 polymerized fatty acid is likewise a distillation product from the 30 polymerization of unsaturated tall oil fatty acids, in the form of a dark very viscous liquid (Gardner color 14) having a 67% trimer acids content and a 33% dimer acids content. UNIDYME™ 60 polymerized fatty acid has an acid number of 192. EXXAL™ 13 iso-tridecyl alcohol is from ExxonMobil Chemical Company (Houston, TX; @exxon.com/exxonchemical). Neopentyl glycol is 35 from Eastman Chemicals (Kingston, TN), FASCAT™ organometallic catalysts, e.g., FASCAT™ 2001 stannous oxalate, are available from Atofina North

America Inc. (Philadelphia, PA: @.elf-atochem.com). HFSC (High Flow Super Cell) is a diatomaceous earth filter aid. The term "AN" refers to acid number, which is synonymous with "acid value" and may be measured by techniques well known in the art. See, e.g., ASTM D 465 (1982). Viscosity measurements are performed according to ASTM D 445 (1996) (Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids). Pour point is determined according to ASTM D 97 (1985) (Pour Point of Petroleum Oils). Flash and Fire Points are measured according to ASTM D 92 (1997) (Standard Test Methods for Flash and Fire Points by Cieveland Open Cup).

10

EXAMPLES

EXAMPLE 1

TRIMER ACID. MONOALCOHOLAND 5 WT% NEOPENTYLGLYCOL

15 A 3-liter flask was charged with UNIDYME™ 60 polymerized fatty acids (1000 g. 3.35 eg.), EXXALTM 13 iso-tridecyl alcohol (568 g. 2.87 eg.) and neopentyl glycol (5.0% based on UNIDYME™ 60 polymerized fatty acids, 50.0 grams, 0.96 eg.). The mixture was heated to 101°C and FASCAT™ 2001 catalyst (0.025% based on UNIDYME™ 60 polymerized fatty acids, 0.25 g) was 20 added. The reaction temperature was increased to a maximum of 220°C and the acid value of the reaction mixture was determined to be 54.9. The reaction mixture was maintained at 220°C for 111/2 hours and vacuum was slowly applied to the mixture to maintain reflux when necessary. The vacuum was then lowered to 10 mm to remove any remaining volatile materials from the reaction 25 mixture. The reaction mixture was then cooled to 130°C and filtered through HFSC using Whatman #1 paper (under vacuum) to give a product with an acid value of 1.1, a Gardner color of 14-, a viscosity at 40°C of 516.8 cSt, a viscosity at 100°C of 44.1 cSt, a viscosity index of +138, a pour point of -33°C, a flash point of 580°F, and a fire point of 650°F.

30

EXAMPLE 2

TRIMER ACID, MONOALCOHOL AND 3.5 WT% NEOPENTYLGLYCOL

A 5-liter flask was charged with UNIDYME™ 60 polymerized fatty acids (1703 g.5.7 eq.), EXXAL™ 13 iso-fridecyl alcohol (1357 g. 6.85 eq.), and neopentyl glycol (5% based on UNIDYME™ 60 polymerized fatty acids, 59.6 g, 1,15 eq.). The mixture was heated to 107°C and FASCAT™ 2001 catalyst

(0.025% based on UNIDYME™ 60 polymerized fatty acids, 0.43 g) was added. The reaction temperature was increased to a maximum of 220°C and the acid value of the reaction mixture was determined to be 40.7. The reaction mixture was maintained at 220°C for 5½ hours and vacuum was slowly applied to the mixture to maintain reflux when necessary. The vacuum was then lowered to 1 mm to remove any remaining volatile materials from the reaction mixture. The reaction mixture was cooled to 130°C and filtered through HFSC using Whatman #1 paper (under vacuum) to give a product with an acid value of less than 2.0 and a viscosity at 40°C of 367cSt.

EXAMPLE 3

10

TRIMER ACID, MONOALCOHOL AND 2.5 WT% NEOPENTYLGLYCOL

A 3-liter flask was charged with UNIDYME™ 60 polymerized fatty acids (1054 g, 3.63 eq.), EXXAL™ 13 iso-tridecyl alcohol (598.7 g, 3.02 eq.)

15 and neopentyl glycol (2.5% based on UNIDYME™ 60 polymerized fatty acids, 26.4 g, 0.51 eq.). The mixture was heated to 117°C and FASCAT™ 2001 catalyst (0.05% based on UNIDYME™ 60 polymerized fatty acids, 0.51 g) was added. The reaction remperature was increased to 220°C and the acid value of the reaction mixture was determined be 30.4. The reaction mixture was maintained at 220°C for 11 hours and vacuum was slowly applied to the mixture to maintain reflux when necessary. The vacuum was then lowered to remove any remaining volatile materials from the reaction mixture. The reaction mixture was cooled to 130°C and filtered through HFSC using Whatman #1 paper (under vacuum) to give a product with a viscosity at 40°C of 365.9 cSt, a viscosity at 100°C of 33.7, a viscosity index of 132, a specific gravity of 0.909 and a Gardner color of 14+.

EXAMPLE 4

BLEACHED TRIMER ACID, MONOALCOHOL AND 5 WT% NEOPENTYLGLYCOL

ackls (800 g, 2.0 eq.) that had been previously bleached with palladium catalyst, ackls (800 g, 2.0 eq.) that had been previously bleached with palladium catalyst, EXXAL™ Iso-tridecyl alcohol (316 g, 1.6 eq.) and neopentyl glycol (5.0% based on UNIDYME™ 60 polymerized fatty acids, 30 g, 0.58 eq.). The mixture was heated to 113°C and FASCAT™ 2001 catalyst (0.025% based on UNIDYME™ 60 polymerized fatty acids, 0.15 g) was added. The reaction temperature was increased to 220°C and the acid value of the reaction mixture was determined to

be 38.6. The reaction mixture was maintained at 220°C for 9½ hours at which point vacuum was applied (to 1 mm) to remove any remaining volatile materials. The reaction mixture was cooled to 130°C and filtered through HFSC using Whatman #1 paper (under vacuum) to give a product with a viscosity at 40°C of 554 cSt, an acid value of 2.4, a Gardner color of 8-, a pour point of -33°C, a flash point of 565°F and a fire point of 655°F.

EXAMPLE 5

DIMER/TRIMER ACID. MONOALCOHOL AND 5% NEOPENTYLGLYCOL

A 3-liter flask was charged with UNIDYME™ 40 polymerized fatty 10 acids (1041 a. 3.53 ea.), EXXAL™ iso-tridecyl alcohol (601 a. 3.04 ea.) and neopentyl glycol (5% based on UNIDYME™ 40 polymerized fatty acids, 52.1 g. 1.0 eg.). The mixture was heated to 113°C and FASCAT™ 2001 catalyst (0.025% based on UNIDYME™ 40 polymerized fatty acids, 0.26 g) was added. 15 The reaction temperature was increased to 220°C and the acid value of the reaction mixture was determined to be 63.6. The reaction mixture was maintained at 220°C for 13 hours and vacuum was slowly applied to the mixture to maintain reflux when necessary. The vacuum was then lowered to 1 mm to remove any remaining volatile materials from the reaction mixture. The reaction 20 mixture was cooled to 130°C and filtered through HFSC using Whatman #1 paper (under vacuum) to give a product with a viscosity at 40°C of 462 cSt, a viscosity of 100°C of 40.5 cSt. a viscosity index of 135, a Gardner color of 13+, an acid value of 0.6, a % moisture of 0.06, a pour point of -30°C, a flash point of 580°F, a fire point of 660°F and a specific gravity of 0.914.

FXAMPI F 6

25

DIMER/TRIMER ACID, MONOALCOHOL AND 25% NEOPENTYLGLYCOL

A 3-liter flask was charged with UNIDYME™ 40 polymerized fatty acids (1037 g, 3.62 eq.), EXXAL™ 13 iso-tridecyl alcohol (716.8 g, 3.62 eq.) and neopentyl glycol (2.5% based on UNIDYME™ 40 polymerized fatty acids, 25.9 g, 0.5 eq.). The mixture was heated to 101°C and FASCAT™ 2001 catalyst (0.05% based on UNIDYME™ 40 polymerized fatty acids, 0.52 g) was acided. The reaction temperature was increased to 220°C and the acid value of the reaction mixture was maintained at 220°C for 11 hours and vacuum was slowly applied to the mixture to maintain reflux when necessary. The vacuum was then lowered to 2 mm to

remove any remaining volatile materials from the reaction mixture. The reaction mixture was cooled to 130°C and filtered through HFSC using Whatman #1 paper (under vacuum) to give a product with a viscosity at 40°C of 368.2 cSl. a viscosity of 100°C of 33.7 cSl, a viscosity index of 135, a pour point of –30°C, a flash point of 580°F, and a fire point of 670°F.

EXAMPLE 7

UNIDYME 40, 2-ETHYLHEXANOL AND 5% NEOPENTYLGLYCOL

A 3-liter flask was charged with UNIDYME™ 40 polymerized fatty (5.0% based on UNIDYME™ 40 polymerized fatty acids (886 g, 3 eq.), 2-ethylhexano (935 g, 2.68 eq.), and neopentyl glycol (5.0% based on UNIDYME™ 40 polymerized fatty acids, 44.3 g, 0.85 eq.). The mixture was heated to 110°C and FASCAT™ 2001 catalyst (0.025 % based on UNIDYME™ 40 polymerized fatty acids, 0.22 g) was added. The reaction temperature was increased to 220°C and the acid value of the reaction mixture was determined to be 63.9. The reaction mixture was maintained at 220°C for 7 hours and vacuum was slowly applied to the mixture to maintain reflux when necessary. The vacuum was then lowered to 10 rmn to remove any remaining volatite materials from the reaction mixture. The reaction mixture was cooled to 130°C and filtered through HFSC using Whatman #1 paper (under vacuum) to give a product with a viscosity at 40°C of 333.9 cSt, a viscosity of 100°C of 33.7 cSt, a viscosity index of 143, an acid value of 2.3, a Gardner color of 15, a pour point of −39°C, a flash point of 590°F, and a fire point of 685°F.

FXAMPLE 8

UNIDYME 40, 2-ETHYLHEXANOL AND 5% NEOPENTYLGLYCOL

25

A 5-liter flask was charged with UNIDYME™ 40 polymerized fatty acids (1933 g, 6.55 eq.), 2-ethylhexanol (732 g, 5.6 eq.), and neopentyl glycol (5% based on UNIDYME™ 40 polymerized fatty acids, 96.7 g, 1.9 eq.). The mixture was heated to 110°C and FASCAT™ 2001 catalyst (0.025 % based on UNIDYME™ 40 polymerized fatty acids, 0.48 g) was added. The reaction temperature was increased to 220°C and the acid value of the reaction mixture was determined to be 45.5. The reaction mixture was maintained at 220°C for 6 hours and vacuum was slowly applied to the mixture to maintain reflux when necessary. The vacuum was then lowered to 1 mm to remove any remaining volatile materials from the reaction mixture. The reaction mixture was cooled to 130°C and filtered through HFSC using Whatman #1 paper (under vacuum) to

give a product with a viscosity at 40° C of 321.5 cSt, an acid value of 2.1, and a Gardner color of ca. 13.

EXAMPLE 9

UNIDYME 40, 2-ETHYLHEXANOL AND 10% NEOPENTYLGLYCOL

A 3-liter flask was charged with UNIDYME™ 40 polymerized fatty acids (1163 g, 3,94 eq.), 2-ethylhexancl (255 g, 2.04 eq.) and neopentyl glycol (10% based on UNIDYME™ 40 polymerized fatty acids, 116.3 g, 2.24 eq.). The mixture was heated to 111°C and FASCAT™ 2001 catalyst (0.025% based on UNIDYME™ 40 polymerized fatty acids, 0.29 g) was added. The reaction temperature was increased to 220°C and the acid value of the reaction mixture was determined to be 47.7. The reaction mixture was maintained at 220°C for 12 hours and vacuum was slowly applied to the mixture to maintain reflux when necessary. The vacuum was then lowered to 2 mm to remove any remaining volatile materials from the reaction mixture. The reaction mixture was cooled to 130°C and filtered through HFSC using Whatman #1 paper (under vacuum) to give a product with a viscosity at 40°C of 1520 cSt.

EXAMPLE 10

20 ESTER BLEND

5

The ester from Example 2 (75% by weight) was combined with the ester from Example 1 (25% by weight) to give an ester blend with the following characteristics: an acid value of 1.3, a viscosity at 40°C of 403.6 cSt, a viscosity at 100°C of 36.3 cSt, a viscosity index of 134, a hydroxyl number of 1.2, a flash point of 555°F, a fire point of 650°F, a pour point of –33°C, a Gardner color of 13-, and an iodine value of 10.4.

EXAMPLE 11 ESTER BLENDS

30 Three blends were prepared as identified in the Table below. The viscosity of each blend was also determined.

Wt % Ester of Example 7	Wt % Ester of Example 5	Viscosity at 40°C (cSt)	
80	20	351.7	
60	40	377.0	
40	60	403.0	

EXAMPLE 12 ESTER BLEND

The ester from Example 8 (40% by weight) and the ester from Example 5 (60% by weight) were combined to give a final product with a viscosity at 40°C of 399 cSt, a viscosity at 100°C of 37.2 cSt, a viscosity index of 138, an acid value of 1.3, a Gardner color of 13, a flash point of 580°F, a fire point of 670°F and a pour point of –33°C.

EXAMPLE 13

A blend was prepared from the ester of Example 8 (80% by weight), and the ester of Example 5 (20% by weight). The final product had a viscosity at 40°C of 349 cSt, a viscosity at 100°C of 34.5 cSt, a viscosity index of 142, an acid value of 1.7, a Gardner color of 13, a flash point of 580°F, a fire point of 675°F, and a pour point of -33°C.

EXAMPLE 14

ESTER-POLYPROPYLENE COMPATIBILITY

20 The compatibility of ester blends of the present invention with polypropylene was determined by measuring the extent to which ester absorbed into polypropylene when the two materials were maintained in contact for several weeks. More specifically, samples of polypropylene extruded strips having dimensions 0.7 mm thickness and 20 mm width were weighed (each strip weight approximately 3 grams). A known weight of esters, in the range of 30 grams, was added to an aluminum pan of dimensions 63.5 mm x 17.5 mm. An individual strip of polypropylene was placed in the aluminum pan with the esters, and the pan was covered with aluminum foil. The sample was maintained at 80°C for a predetermined period.

10

First Ester	Second Ester	% Wt Gain Wk 1	% Wt Gain Wk 2	% Wt Gain Wk 4
80% Example 7	20% Example 5	4.95	5.06	4.93
60% Example 7	40% Example 5	4.23	4.88	4.88
40% Example 7	60% Example 5	4.56	5.09	4.90

For comparison, when the same procedure was followed using poly-alpha olefins instead of esters, the % Wt Gain values were 4.59 (1 week), 5.43 (2 weeks) and 5.94 (4 weeks).

All publications and patent applications mentioned in this specification are herein incorporated by reference to the same extent as if each individual publication or patent application was specifically and individually incorporated by reference.

From the foregoing it will be appreciated that, although specific
embodiments of the invention have been described herein for purposes of
illustration, various modifications may be made without deviating from the spirit
and scope of the invention. Accordingly, the invention is not limited except as
by the appended claims.

CLAIMS

A composition comprising a blend of first and second esters.

the first ester comprising a reaction product of first reactants comprising trimer acid or reactive equivalent thereof, first polyhydric alcohol or reactive equivalent thereof, and first monohydric alcohol or reactive equivalent thereof, the first monohydric alcohol being selected from the group of $C_{\text{B-18}}$ monohydric alcohols or reactive equivalents thereof.

the second ester comprising a reaction product of second reactants comprising trimer acid or reactive equivalent thereof, second polyhydric alcohol or reactive equivalent thereof, and second monohydric alcohol or reactive equivalent thereof; the second monohydric alcohol being selected from the group of C₆₋₁₀ monohydric alcohols or reactive equivalents thereof;

the first and second monohydric alcohols being non-identical.

- 2. The composition of claim 1 wherein the first reactants and the second reactants each further comprise dimer acid.
- The composition of claim 2 wherein the first reactants and the second reactants each comprise dimer acid and trimer acid, in a dimer acid:trimer acid weight ratio of 20:80 to 80:20, the weight ratio independently selected in each of the first and second reactants.
- The composition of any of claims 1-3 wherein the trimer acid is hydrogenated trimer acid.
- The composition of any of claims 1-4 wherein the first monohydric alcohol is selected from C₁₀₋₁₄ primary monohydric alcohols.
- 6. The composition of any of claims 1-5 wherein the first monohydric alcohol is *iso*-tridecyl alcohol.
- $7. \qquad \text{The composition of any of claims 1-6 wherein the first and second polyhydric alcohols are each selected from C_{3-15} polyhydric alcohols.}$

 The composition of any of claims 1-6 wherein the first and second polyhydric actoohs are each independently selected from pentaerythritol, tri-pentaerythritol, tri-pentaerythritol, trimethylolpropane, ethylene glycol and neppentlyl divcol.

- The composition of any of claims 1-6 wherein the first and second polyhydric alcohols are each neopentyl glycol.
- The composition of any of claims 1-6 wherein the second monohydric alcohol is selected from 2-ethylhexanol, 2-octanol and cyclohexyl alcohol.
- The composition of any of claims 1-6 wherein the second monohydric alcohol is 2-ethylhexanol.
- 12. The composition of any of claims 1-11 wherein the first ester has a greater viscosity than the second ester.
- \$13.\$ The composition of any of claims 1-12 having a viscosity of 30-50 cSt at 100°C.
- The composition of any of claims 1-13 having a viscosity of about 40 cSt at 100°C.
- 15. The composition of claim 1 wherein the first monohydric alcohol is selected from the group of C₁₉₋₁₉ primary monohydric alcohols; the second monohydric alcohol is selected from the group consisting of C₂ monohydric alcohols; the first and second polyhydric alcohols are selected from C₂₋₁₉ polyhydric alcohols, and the viscosity of the composition is in the range of 30-50 cSt at 100°C.
- 16. A method for preparing an ester composition having a viscosity in the range of 30-50 cSt at 100°C, the method comprising the steps of:
- preparing a first ester, the first ester comprising a reaction product of first reactants comprising trimer acid. first polyhydric alcohol and first.

monohydric alcohol; the first monohydric alcohol selected from the group of C_{g. 18} monohydric alcohols;

- b) preparing a second ester, the second ester comprising a reaction product of second reactants comprising trimer acid, second polyhydric alcohol and second monohydric alcohol; the second monohydric alcohol selected from the group of Govern monohydric alcohols; and
- blending the first and second esters together in a proportion to provide an ester composition having a viscosity in the range of 30-50 cSt at 100°C;

with the proviso that the first and second monohydric alcohols are non-identical.

- 17. The method of claim 16 wherein the first reactants and the second reactants each further comprise dimer acid, in a dimer acid:trimer acid weight ratio of 20:80 to 80:20, the weight ratio independently selected in each of the first and second reactants.
- The method of any of claims 16-17 wherein the trimer acid is hydrogenated trimer acid.
- The method of any of claims 16-18 wherein the first monohydric alcohol is selected from C₁₀₋₁₄ primary monohydric alcohols.
- 20. The method of any of claims 16-19 wherein the first and second polyhydric alcohols are each selected from C₃₋₁₅ polyhydric alcohols.
- The method of any of claims 16-20 wherein the second monohydric alcohol is selected from 2-ethylhexanol, 2-octanol and cyclohexyl alcohol.
- 22. The method of any of claims 16-21 wherein the first ester has a greater viscosity than the second ester.
- $23. \quad \text{The method of claim 16 wherein the first monohydric alcohol is selected from the group of C_{10-15} primary monohydric alcohols; the second monohydric alcohol is selected from the group consisting of $C_8$$

monohydric alcohols; the first and second polyhydric alcohols are selected from C_{5-16} polyhydric alcohols; and the first ester has a greater viscosity than the second ester.

- A composition prepared by the method of any of claims 16-
- 25. A cable-filling composition comprising silica and a blend of first and second esters.

the first ester comprising a reaction product of first reactants comprising trimer acid, first polyhydric alcohol and first monohydric alcohol; the first monohydric alcohol selected from the group of C₈₋₁₈ monohydric alcohols;

the second ester comprising a reaction product of second reactants comprising trimer acid, second polyhydric alcohol and second monohydric alcohol; the second monohydric alcohol selected from the group of C₆₋₁₀ monohydric alcohols:

the first and second monohydric alcohols being non-identical.

- 26. The composition of claim 25 wherein the first monohydric alcohol is selected from the group of C₁₉₋₁₉ primary monohydric alcohols; the second monohydric alcohol is selected from the group consisting of C₂ monohydric alcohols; the first and second polyhydric alcohols are selected from C₂₋₁₉ polyhydric alcohols, and the viscosity of the blend is in the range of 30-50 cst at 10°C.
- 27. A method for insulating the contents of a cable, the method comprising
- a) preparing a cable-filling composition comprising silica and a blend of first and second esters; the first ester comprising a reaction product of first reactants comprising trimer acid, first polyhydric alcohol and first monohydric alcohol; the first monohydric alcohol selected from the group of $C_{\delta-1\delta}$ monohydric alcohols; the second ester comprising a reaction product of second reactants comprising trimer acid, second polyhydric alcohol and second monohydric alcohol; the second monohydric alcohol selected from the group of $C_{\delta-1\delta}$ monohydric alcohols; the first and second monohydric alcohols being non-identical:

b) placing the cable-filling composition of step a) into a fiber optic cable.

- 28. The method of claim 27 wherein the cable is a fiber optic cable.
- 29. A cable comprising a cable-filling composition; the cable-filling composition comprising sitica and a blend of first and second esters; the first ester comprising a reaction product of first reactants comprising trimer acid, first polyhydric alcohol and first monohydric alcohol; the first monohydric alcohol selected from the group of C₈₋₁₈ monohydric alcohols; the second ester comprising a reaction product of second reactants comprising trimer acid, second polyhydric alcohol and second monohydric alcohol; the second monohydric alcohol selected from the group of C₈₋₁₉ monohydric alcohols; wherein the first and second monohydric alcohols are non-identical.
- The cable of claim 29 wherein the cable is a fiber optic cable.

Exhibit C

A copy of the May 7, 2007 letter from J.M. Gilbreth to Nancy Mosby.

GILBRETH & ASSOCIATES, P.C.

J. M. (Mark) Gilbreth 124 Mary A. Gilbreth, Ph.D.⁴ Deborah G. Segers 134 Licensed to practice law in ¹TX, ²OK, and ⁵AR ⁴Registered, United States Patent & Trademark Office

May 7, 2007

Nancy D. Mosby 4115 Swarthmore Street Houston, Texas 77005 VIA FEDERAL EXPRESS NO. 860444162800

Re: ARZ - 024630-US (Application No. 10/572,994, PCT/US02/12961) Polybasic Acid Feters and Their Use in Cables

Dear Ms. Mosby,

Our firm represents Arizona Chemical Company in the above-referenced patent matter. Upon review of the files, it has come to our attention that you did not execute a declaration for this matter. We have enclosed a complete copy of the specification for the application listed above. Please sign the attached declaration and power of attorney. Return the executed declaration and power of attorney to us in the Federal Express envelop we have provided for your convenience.

We noted from the files that you previously refused to execute a declaration. If you are still refusing to sign the declaration, please send me an email or sign this letter on the bottom and return in the FEDEX envelope, confirming your continuing refusal to sign the declaration for the above referenced matter.

By Monday, May 14, 2007 please either: (1) send the executed declaration to me or (2) send me an email or letter confirming your refusal to sign.

Thank you for your attention to this matter. If you need further assistance, you can call me at 713/667-1200 or email me at <u>imark@gilbreth.org.</u>

Very truly yours.

J. M. (Mark) Gilbreth

Enclosures

Fed US Airbill	
Express US Airbill Field BLO4 4416 2800	CONTRACTOR OF THE PARTY OF THE
1 From Augusters Johnson 1 Sander's Feder	4a Express Package Service Packages up to 150 lbs. - FedEx Priority Overnight FedEx Standard Overnight FedEx First Overnight
Date 5- 1-0 Account Number 100 Jan 1- J	FedEx Priority Overnight TedEx Priority Overnight TedEx Standard Overnight TedEx Standard Overnight TedEx Standard Overnight TedEx Standard Overnight TedEx Ted Overnight TedEx TedE
Sender's MANK GIBALTH Phone (713) (dad-1200)	FedEx 20ay Second houseas sin; "humain Second houseas sin; "humain Second houseas sin side of the second single side of the second single side of the second side of
Company (OII BRETH & ASSOC, PC	4b Express Freight Service Packages over 150 lbs.
1700 10 1000 Satis # 920	FedEx 1Day Freight* FedEx 3Day Freight FedEx 3Day F
Address 6 750 W. Coop. Conf. Co. Co. Co. Co. Co. Co. Co. Co. Co. Co	** Cast for Confirmation: *** To most focusions. 5 Packaging
city Ball And State 14 ZIP 77 401	FedEx FedEx FedEx FedEx Other Finds under the first share it is not sh
2 Your Internal Billing Reference AR2 - 024630 2105	6 Special Handling wassers for the Street Control of the Street Co
Becomes Naccity Makail	SATURDAY Delivery MOT Available for Feet Standard Overeight. MOT Overeight of The Standard Overeight. MOT Overeight or The Standard Overeight.
Name NANCY VIVOXO Phone (Half Standard Country (C. 1977) - Confirming over 1971 - Confirming over 1971 - Confirming over 1971 - Confirming over 1971 - Country (C. 1971) -
Company	No Yes Separate Decision Of Se
Recipient's 4115 Sunthmore ST.	Dangagus goods (including dry ca) carrox be shipped in FedEx peckaging. Cargo Aircraft Only
Address /// Dept. Front Supplementaries Chapt. Front Supplementaries Chapt	Sander Recipient Third Party Credit Card CastlyCheck
Address To request applicage be hald at a apocalis Fredix location, print Fredix address have.	Ge billed.
Houses IV - Door	Folds No. Dog. Credit Carl St. Total Psycloges Total Weight Total Declared Value?
City COOLOG State & ZIP FROM S	\$.00
	The delations beneath \$150 prices you declare a higher white San buck for details, the ways this Almiller was you to the San buck for details, the ways this Almiller was you to the send of our in facts. General build, be
Store Vigo and ressessate tedex com:	No Signature Required Required Package noting in ordinary Report of trippersor Report of Re
	For delivery. Rev. Date 405-Part #190391-01964-2005 FedEx-PRINTED IN U.S.A. SRY
·	

Page 1 of Express Mail Label No.

Docket No.	
024630	

Declaration and Power of Attorney For Patent Application English Language Declaration

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

POLYBASIC ACID ESTERS AND THEIR USE IN CABLES

the specification of which (check one) is attached hereto.

	03/23/2006	as United States Application No	. or PCT Internationa
• •	ımber <u>10/572,994</u>		
and was amer	nded on		
		(if applicable)	
		understand the contents of the above in amendment referred to above.	dentified specification
		e United States Patent and Trademark bility as defined in Title 37, Code of	
Section 365(b) of	any foreign application	under Title 35, United States Code, n(s) for patent or inventor's certificate	, or Section 365(a) of
Section 365(b) of any PCT Internat States, listed belo patent or inventor application on whice	any foreign applicatio ional application whic w and have also ident is certificate or PCT Int ch priority is claimed.		, or Section 365(a) o other than the Unite foreign application fo
Section 365(b) of any PCT Internat States, listed belo patent or inventor application on which	any foreign applicatio ional application whic w and have also ident is certificate or PCT Int ch priority is claimed.	n(s) for patent or inventor's certificate th designated at least one country of iffed below, by checking the box, any	, or Section 365(a) of their than the Unite foreign application for late before that of the Priority Not Claimed
Section 365(b) of any PCT Internat States, listed belo patent or inventor application on white Prior Foreign Appli	any foreign applicatio ional application whic w and have also ident s certificate or PCT in ch priority is claimed.	n(s) for patent or inventor's certificate h designated at least one country of iffed below, by checking the box, any ternational application having a filing of 	or Section 365(a) on the than the Unite foreign application for the before that of the
Section 365(b) of any PCT Internat States, listed belo patent or inventor application on white Prior Foreign Appli	any foreign applicatio ional application whic w and have also ident is certificate or PCT Int ch priority is claimed.	n(s) for patent or inventor's certificate th designated at least one country of iffed below, by checking the box, any	, or Section 365(a) of their than the Unite foreign application for late before that of the Priority Not Claimed
Section 365(b) of any PCT Internat States, listed belo patent or inventor application on white Prior Foreign Appli Number)	any foreign applicatio ional application whic w and have also ident s certificate or PCT in ch priority is claimed.	n(s) for patent or inventor's certificate h designated at least one country of iffed below, by checking the box, any ternational application having a filing of 	or Section 365(a) bther than the Unite toreign application fo late before that of the Priority Not Claimed
Section 365(b) of any PCT Internat States, listed belo patent or inventor	any foreign applicatio ional application whic wand have also ident s certificate or PCT Int ch priority is claimed. ication(s) (Country)	n(s) for patent or inventor's certificate h designated at least one country of iffed below, by checking the box, any ternational application having a filing of (Day/Month/Year Filed)	, or Section 365(a) of other than the Unite foreign application for late before that of the

I hereby claim the benefit under 35 U.S.C. Section 119(e) of any United States provisional application(s) listed below:

60/286,889	04/27/2001
(Application Serial No.)	(Filing Date)
(Application Serial No.)	(Filing Date)
(Application Serial No.)	(Filing Date)

I hereby claim the benefit under 35 U. S. C. Section 120 of any United States application(s), or Section 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. Section 112, I acknowledge the duty to disclose to the United States Patent and Trademark Office all information known to me to be material to patentability as defined in Title 37, C. F. R., Section 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application:

PCT/US02/12961	04/25/2002	
(Application Serial No.)	(Filing Date)	(Status) (patented, pending, abandoned)
(Application Serial No.)	(Filing Date)	(Status) (patented, pending, abandoned)
(Application Serial No.)	(Filing Date)	(Status) (patented, pending, abandoned)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

POWER OF ATTORNEY: As a named inventor. I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith. (itst name and resistration number)

ATTORNEYS ASSOCIATED WITH CUSTOMER

NUMBER 50947

Send Correspondence to: ADDRESS ASSOCIATED WITH CUSTOMER NUMBER 50947

Direct Telephone Calls to: (name and telephone number)
J. M. (MARK) GILBRETH, 713/667-1200 X102

Full name of sole or first inventor LLOYD A. NELSON

Sole or first inventor's signature

Residence 107 TEAKWOOD DRIVE, SAVANNAH, GA 31410

Citizenship

USA

Post Office Address

SAME AS RESIDENCE

Full name of second inventor, if any NANCY D. MOSBY Second inventor's signature

MAY, 2007

4115 SWARTHMORE STREET, HOUSTON, TX 77005

Citizenship USA

Post Office Address

SAME AS RESIDENCE



IP SERVICES



Home IP Services FatentScope Fatent Search



Search result: 1 of 1

(WO/2002/088208) POLYBASIC ACID ESTERS AND THEIR USE IN CABLES

Biblio, Data

Description

Claims

National Phase

Notices

Documents

Latest bibliographic data on file with the International Bureau

Publication Number: WO/2002/088208

International Application No.: PCT/US2002/012961 International Filing Date:

07.11.2002 Publication Date: Chapter 2 Demand Filed: 14.11.2002

25.04.2002

Int. Class.:

C08L 67/00 (2006.01), G02B 6/44 (2006.01), C08K 3/36 (2006.01)

Applicants:

ARIZONA CHEMICAL COMPANY [US/US]; Suite 500 4600 Touchton Road Jacksonville, FL 32246-8225 (US) (All Except US).

NELSON, Lloyd, A. [US/US]; 107 Teakwood Drive Savannah, GA 31410-1626 (US) (US Only). MOSBY, Nancy, D. [US/US], 1619 Wilmington Island Road Savannah, GA 31410-4519 (US) (US Only).

Inventors: Agent:

NELSON, Lloyd, A. [US/US]; 107 Teakwood Drive Savannah, GA 31410-1626 (US).

MOSBY, Nancy, D. [US/US]; 1619 Wilmington Island Road Savannah, GA 31410-4519 (US). PARKER, David, W.: Seed Intellectual Property Law Group PLLC Suite 6300 701 Fifth Avenue Seattle,

WA 98104-7092 (US).

Priority Data: 60/286,880 27.04.2001 US

Title: POLYBASIC ACID ESTERS AND THEIR USE IN CABLES

Abstract:

A composition comprising a blend of first and second esters; the first ester comprising a reaction product of first reactants comprising trimer acid, first polyhydric alcohol and first monohydric alcohol; the first monohydric alcohol selected from the group of C8-18 monohydric alcohols; the second ester comprising a reaction product of second reactants comprising trimer acid, second polyhydric alcohol and second monohydric alcohol; the second monohydric alcohol selected from the group of C6-10 monohydric alcohols; the first and second monohydric alcohols being non-identical. This blend of esters, in combination with silica, is useful as a sealant composition, e.g., as a cable-protectant composition.

Designated States:

AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZM, ZW.

African Regional Intellectual Property Org. (ARIPO) (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG,

Eurasian Patent Organization (EAPO) (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM)

European Patent Office (EPO) (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, African Intellectual Property Organization (OAPI) (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR,

NE. SN. TD. TG).

Publication Language:

English (EN)

Filing Language:

English (EN)



IP SERVICES



-one If Services FatentScope Patent Search



Search result: 1 of 1

(WO/2002/088208) POLYBASIC ACID ESTERS AND THEIR USE IN CABLES

Biblio, Data Description Claims

National Phase

Notices

Documents

POLYBASIC ACID ESTERS AND THEIR USE IN CABLES BACKGROUND OF THE INVENTION Field of the Invention This invention generally relates to protecting moisture-sensitive substrates from moisture, to articles such as a fiber optic cable that contain both a moisture sensitive component and a protectant composition; and to compositions that may be used to protect substrates from moisture and/or corrosion.

Description of the Related Art Transmission cables (e. g., service or power wires/cables) as well as telecommunication devices (e. g., fiber optic cables, telephone cables, terminal blocks, junctions and connections) are often exposed to harsh environmental conditions. Yet, both transmission cables and telecommunication devices are required to maintain uninterrupted mechanical and electrical characteristics despite the surrounding environmental conditions. Historically, transmission cables have been strung above ground between poles. Increasingly, these cables are being buried beneath the ground for aesthetic reasons and to protect them from extremes of temperature, rain, snow, ice, high winds, falling tree limbs and the like. However, even when placed underground, these cables are subject to many environmental stresses including mechanical shock during back filling, moisture, the possibility of water immersion, attack from rodents and exposure to salt and other corrosive materials.

Transmission cables typically contain either a bundle of individually insulated copper wires, or a bundle of fiber optic cables. The copper wires, in particular, are often protected by an inner metal shield, which covers the bundle of wires, and an outer sheath made from plastic or other insulating material. Likewise, a communication cable is generally constructed of an outer plastic lacket and an inner metal core wrap with an annular space there between. See, e. g., U. S. Patent No. 3,745,321 to Eager, Jr. et al. The core wrap enwraps a plurality of twisted insulated conductors with a filler material, such as petroleum, wax, or other hydrocarbons, located between the conductors. The filler material typically functions as an insulator.

Even though wires and cables are normally well protected, if the protective outer sheath is cut or otherwise broken, water can seep into the wire or cable. Whenever moisture is present in the interior of a cable or wire, it tends, over a period of time, to migrate or flow longitudinally into connections at the splice closures, terminals, or the like. In the special case of optical fibers, passage of the water to connection points or terminals and associated equipment will typically result in damage to such equipment, especially to any metal parts thereof, and can also cause problems at low temperature or freezing environments due to fiber microbending. This is particularly true when an immersed wire is cut

In an inadequately protected device, corrosion of the contacts or short circuits caused by moisture or other outside elements will eventually interrupt service. Replacement or repair of the cables, whether strung above the ground or buried, can be difficult and/or expensive, and the interruption of service may be particularly irritating to the customer. Because of this, much attention has focused on the protection of these cables from both physical damage and from chemical attack. As a result, various techniques have been disclosed to prevent or restrict migration of moisture along the internal passages of a cable.

In one such technique, cable passages may be filled by a pressurized gas. Upon escape of gas through a ruptured cable jacket, an alarm provides notice of cable damage. However, the gas does not prevent the flow of water into and along the cable internal passages before the repair is effected.

U. S. Patent Nos. 4,867,526; 5,082,719; and 5,163,115 disclose a super-absorbent polymer or tape which is impregnated with a superabsorbent material for preventing water migration through cables. It has been found that, for a number of reasons, the production and/or installation of these materials is often impractical and/or uneconomical.

Silicone grease lubricant, elastomeric seals, and/or mastic sealing strips have been used in an attempt to seal out moisture. While these sealants offer some protection, moisture can often seep into the enclosure and cause a short circuit, which will interrupt telecommunications service, particularly when the terminal block is buried underground.

It is also known that insulating materials, such as mineral oil, protect the conductor from moisture and corrosive materials. However, these such insulating materials drip when the cable is being serviced (i. e. cut). To overcome this dripping problem, various gelling agents are known to be added to the insulator, to thereby provide a gellied, non-dripping protectant composition. See, e. a., U. S. Patent Nos. 6, 169, 160 (-5, 160, 39); and 6, 085, 009

Zeolites have recently been suggested as a suitable material to impart moisture-resistance to a cable. See, e.g., US 6 205 276 B1.

Hydrophobic fumed silica has been used as a gelling agent, particularly for fiber optic cables. See e. g., U. S. Patent Nos. 5,905.833, 5,902.849, 5,737,469, 5,285,513, 5,276,757, and 5,187,763: and European Patent EP 0 206 234 B1. The furned silica, in combination with a suitable fluid, will cause the fluid to become gelled and/or demonstrate thixotropic properties. Which is advantageous for a cable protectant composition. Furned silica is rather expensive and so suitable fluids that are relatively less expensive have a commercial advantage.

Due to the relatively long lengths of cable that are typically installed, certain cable components are made from plastic components such as polyproplien in an attempt to both reduce the cost of the cable as well as obtain multiple supply sources for such components. This presents a problem to the supplier of protectant compositions in that many known protectant compositions are not compatible with plastics such as polyproplyene and can cause deterioration of those materials. Furthermore, aithough only a fairly small amount of protectant composition is present in a cross-section of cable, because the cables are extremely long, even a small difference in the per pound cost of the protectant composition can have a large commercial advantage in the marketplace, assuming all other performance properties are pet

A need therefore exists for suitable and cost-effective protectant compositions to be used in transmission cables and telecommunication devices. The present invention fulfills these needs and further provides related advantages

SUMMARY OF THE INVENTION In one aspect, the present invention provides a composition comprising a blend of inst and second esters: the first ester comprises a reaction product of first reactants; the first reactants comprise trimer acid. first polyhydric alcohol and first monohydric alcohol; the first monohydric alcohol is selected from the group of C8-18 monohydric alcohols; and the second ester comprises a reaction product of second reactants; the second reactants comprise trimer acid, second polyhydric alcohol and second monohydric alcohol; the second monohydric alcohol is selected from the group of C610 monohydric alcohols. The first and second monohydric alcohols are nonintentical

In another aspect, the present invention provides a method for preparing an ester composition having a viscosity in the range of 30-50 cSt (centistokes) at 100°C. The method comprises a) preparing a first sets: the first ester comprising a reaction product of first reactants, the first reactants comprising timer acid, first polyhydric alcohol and first monohydric alcohol is selected from the group of C8-18 monohydric alcohols; b) preparing a second ester, the second ester comprising a reaction product of second reactants, the second reactants comprising timer acid, second polyhydric alcohol and second monohydric alcohol, where the second monohydric alcohols; and c) blending the first and second ester dispersion of the proportion to provide an ester composition having a viscosity in the range of 30-50 cSt at 100°C; with the proviso that the first and second monohydric alcohols are non-identified.

The present invention also provides a composition prepared by the above-described method.

In another aspect, the present invention provides a cable-filling composition comprising silica and a blend of first and ascond esters. The first esteral comprises a reaction product of first reactants, the first reactants comprising trimer acid, first polyhydric alcohol and first monohydric alcohol, where the first monohydric alcohol is selected from the group of C81 a monohydric alcohols. The second ester comprises a reaction product of second reactants, the second reactants comprising trimer acid, second polyhydric alcohol and second monohydric alcohol, where the second monohydric alcohol is selected from the group of C810 monohydr

The first and second monohydric alcohols are non-identical.

In another aspect, the present invention provides a method for insulating the contents of a cable, e.g., a fiber optic cable. The method comprises a preparing a cable-filling composition comprising sities and a behalf of first and second extension in the cable of the cable of the second extension product of first reactants, the first reactants comprising timer acid, first exclusion and first monohydric alcohol where the first monohydric alcohol is selected from the group of C8-18 monohydric alcohols; it he second extension and first monohydric alcohols is expected from the group of C8-18 monohydric alcohols where the first monohydric alcohol where the second monohydric alcohol is selected from the group of C8-10 monohydric alcohols; the first and second monohydric alcohols being non-identical; and by ladicing the cable-filling composition of steps in jinto a cable, e.g., a filter ordic cable.

The present invention also provides a fiber optic cable comprising a cable-filling composition: the cable-filling composition comprising silica and a blend of first and second esters; the first ester comprising a reaction product of first reactants comprising timer each, first polyhydric alcohol and first monthydric alcohol; selected from the group of Cg-18 monohydric alcohols; the second ester comprising a reaction product of second reactants comprising timer act, second polyhydric alcohol and second monohydric alcohol; the second monohydric alcohol selected from the group of C610 monohydric alcohols; wherein the first and second monohydric alcohols are non-identical.

These and other aspects of the present invention are described in further detail below.

DETALED DESCRIPTION OF THE INVENTION In one aspect, the present invention provides a composition comprising a blend of first and second selsers, wherein the first sets comprises a reaction product of first reactants comprising timer acid or a reactive equivalent thereof, first polyhydric alcohol or reactive equivalent thereof. First polyhydric alcohol or reactive equivalent thereof the first monohydric alcohol or preactive equivalent thereof. The second ester comprises a reaction product of second reactants comprising timer acid or reactive equivalent thereof, second polyhydric alcohol or reactive equivalent thereof, second polyhydric alcohol or reactive equivalent thereof, second polyhydric alcohol or reactive equivalent may be a second monohydric alcohol being selected from the group of C610 schools. The first and second monohydric alcohol being selected from the group of C610 schools. The first and second monohydric alcohol ser non-identical. Set/ore further describing these compositions of the present invention, the reactants used to prepare the components thereof will be discussed.

Unsaturated fatty acids, as obtained from, e.g., vegetable oils, tallow, and tall oil (the latter being known as tall oil fatty acids, art OFA) may be subjected to thermal opolymerization, typically in the presence of a clay catalyst, to provide a product known commercially as polymerized fatty acid. It oa large extent, the polymerized fatty acid is the dimerization and trimerization product of the fatty acid. Because these fatty acids kpically contain 18 carbons, the corresponding dimerized polymerized fatty acids, also called dimer acid, contains 36 carbon atoms. The corresponding trimerized polymerized fatty acids, also called timer acid, contains 54 carbon atoms. The dimer acids are a mixture of complicated isomeric structures. Detailed descriptions of polymerized fatty acid, in terms of product, composition and uses, may be found in, for example, Naval Stores-Production, Chemistry and Utilization, D. F.

Zinkel and J. Russel (eds.), Pulp. Chem. Assoc. Inc., 1989, Chapter 23 and the Encyclopedia of Chemical Technology II, Vol. 8, pages 847-848. See also, U. S.

Patent No. s 2.904.415; and 2.482.761, and Industrial and Engineering Chemistry, 32; 802 (1940).

The polymerization of fatty acids typically provides a mixture of dimer and and trimer acid. These two materials have different boiling points and can be separated from one another by distillation. For instance, dimer can be distilled away from other polymerized fatty acids at a temperature ranging from 250-280°C (at 0.3 to 0.5 mm·Hg) while trimers may be distilled from other polymerized fatty acids at a temperature ranging from 280-301°C (at 0.05 to 0.0 fm·Hg). Accordingly, by suitable distillation a mixture of polymerized fatty acids can provide trimer acid in various purities, also known as crades.

For the present specification, the term'timer said'will be used to designate a composition containing polymerized carboxylic acid which is at least 20% trimer based on the total weight of the polymerized fatty acid in the composition. Typically, it is difficult to refine trimer acid to a purity of greater than 80%. Accordingly, a preferred trimer acid of the present invention is contained within a mixture of polymerized fatty acids, where the polymerized fatty acids also includes dimer acid, and the weight ratio of dimer acid: timer acid ranges from 20: 80 to 80: 70.

A reactive equivalent of trimer acid is a material that may be used in place of trimer acid to provide essentially the same seter product in the esterification reaction of the present invention. Hydrogenated trimer acid is a reactive equivalent of trimer acid. As initially formed, dimer acid and trimer acid typically contain unsaturated polybasic acids. This unsaturation may, to some degree, be eliminated by subjecting the dimer acid/trimer acid to hydrogenation under suitable reaction conditions whereby the unsaturation reacts with hydrogen and becomes saturated. Trimer acid that has been subjected to a hydrogenation process is commonly referred to as hydrogenated trimer acid, and is well known in the naval stores industry. Hydrogenated trimer acid and side acid. Hydrogenated trimer acid and side in acid. Hydrogenated trimer acid, and thus its use in an esterification process provides lighter colored esters. In addition, esters prepared from hydrogenated trimer acid, and thus its use in an esterification process provides lighter colored esters. In addition, esters prepared from hydrogenated trimer acid.

Accordingly, hydrogenated trimer acid is a preferred trimer acid for use in the present invention.

Another reactive equivalent of trimer acid is a partial or complete esterification product of trimer acid. For example, trimethyl trimerate and triethyl trimerate are esterification products of trimer acid that may be used in the present

invention. When employed in the present invention, a trimerate ester of trimer acid will effectively react to provide the same product as is obtained from trimer acid itself. The esterification product will, however, generate methanol or ethanol, or whatever other alcohol was used to esterify the trimer acid. This alcohol is preferably of low molecular weight and/or high volatility so that it can be readily distilled from the reaction mixture during preparation of the composition of the present invention. Another reactive equivalent is the acid halide of trimer acid

Many companies currently manufacture and sell polymerized fatty acids that include trimer acid according to the present invention. For example, Arizona Chemical (Jacksonville, FL; &commat, arizona chemical, com) sells UNIDYMEX 40 dimer acid that contains about 35% trimer acid and 65% dimer acid, and UNIDYMEX 60 trimer acid that contains about 67% trimer acid and 33% dimer acid. Additional current suppliers of trimer acid and trimer acidcontaining polymerized fatty acids include Henkel Corporation, Emery Oleochemicals Division (Cincinnati, OH. with their EMPOLTM line of dimer and trimer acids) and Unigema North America (Wilmington, DE, with their PRIPOLTM line of dimer and trimer acids).

In one aspect, each of the first reactants and the second reactants further comprise dimer acid. For example, the first reactants and the second reactants each may comprise dimer acid and trimer acid, in a dimer acid: trimer acid weight ratio of 20: 80 to 80: 20, the weight ratio independently selected in each of the first and second reactants. In one aspect, at least one of the reactants employs a blend of dimer acid and trimer acid wherein the trimer acid is present at a greater weight percent, i. e., greater than 50 wt%, than the dimer acid.

The polyhydric alcohol, which may also be referred to as a polyol, has two or more hydroxyl groups. Thus, the polyol has of the chemical formula R\OH) n wherein R'is an n-valent organic group. Optionally, R1 is a C2-C20 organic group without hydroxyl substitution, and preferably R1 is a Cs-Cis organic group. Suitable polyhydric alcohols include, without limitation, ethylene glycol, propylene glycol, neopentyl glycol, butyleneglycol, glycerol, trimethylolpropane. pentaerythritol, tris (hydroxylmethyl) methanol, di-pentaerythritol, and tri-pentaerythritol. In one aspect of the invention, the first and second polyhydric alcohols are each independently selected from pentaerythritol, di-pentaerythritol, tripentaerythritol, trimethylolpropane, ethylene glycol and neopentyl glycol. In one aspect, at least one of the first and second polyhydric alcohols is neopentyl glycol (CAS No. 126-30-7), also known as 2,2-dimethyl-1, 3-propanediol, while in a preferred embodiment the first and second polyhydric alcohols are each neopentyl glycol. Again, a reactive equivalent could be used, e. g., the acetate ester of the polyol.

The monohydric alcohol has a single hydroxyl group. Thus, the monohydric alcohol may be represented by the chemical formula R2-OH where R2 is an organic group containing carbons. The identity of the monoalcohol has a very important impact on the viscosity of the ester produced from the monoalcohol in combination with the polyol and trimer acid. In general, as the molecular weight of the monoalcohol increases, the viscosity of the ester derived therefrom increases.

The present invention provides a blend of first and second esters.

The first ester is prepared from a first monoalcohol while the second ester is prepared from a second monoalcohol.

The first monoalcohol is a C8-18 monohydric alcohol. In other words, the first alcohol is an organic compound having at least 8, and no more than 18, carbon atoms, and a single hydroxyl (OH) group. The first monoalcohol may be represented by the formula R-OH, where R is a hydrocarbon having 8-18 carbons. Suitable examples include 1octanol, 1- decanol, 1-dodecanol (a. k. a. lauryl alcohol), 1-tridecylalcohol, 1-tetradecanol (a. k. a. myristyl alcohol), palmityl alcohol, and stearyl alcohol. The first alcohol may be linear or branched. In one aspect of the invention, the first alcohol is a branched alcohol. A preferred branched alcohol is iso-tridecyl alcohol, available from ExxonMobil Chemicals as EXXALTM 13 alcohol. In another aspect, the first monohydric alcohol is selected from C1014 primary monohydric alcohols.

The second monoalcohol is a C610 monohydric alcohol. In other words, the second alcohol is an organic compound having at least 6, and no more than 10, carbon atoms, and a single hydroxyl (OH) group. The second monoalcohol may be represented by the formula R3-OH, where R3 is a hydrocarbon having 6-10 carbons. The second alcohol may be linear, branched, or cyclic. Suitable examples include 1-hexanol, 1-heptanol, 1-octanol, 1-nonanol, 1-decanol, 2hexanol, 2-heptanol, 2-octanol (a. k. a. capryl alcohol), 2-nonanol, 2- decanol, cyclohexanol, cycloheptanol, and cyclooctanol. In one aspect of the invention, the first alcohol is a branched alcohol. A preferred branched alcohol is 2ethylhexanol. In another aspect, the second monohydric alcohol is selected from 2-ethylhexanol, 2-octanol and cyclohexyl alcohol

Both monohydric and polyhydric alcohols useful in the present invention are very well known in the art and may be obtained from many commercial suppliers including, for example, Aldrich (Milwaukee, WI; @aldrich, sial. com); EM Industries, Inc. (Hawthorne, NY; @ emscience.com); Lancaster Synthesis, Inc. (Windham, NH: lancaster, co. uk); and Spectrum Quality Product, Inc. (New Brunswick, NJ; @spectrumchemical, com).

The first ester comprises the reaction product of reactants comprising trimer acid, first polyhydric alcohol and first monohydric alcohol or reactive equivalents of one or more. This first ester has a low acid number, that is, an acid number of less than 25, preferably less than 20.15.10.5.2, or 1. It has a viscosity, as measured in centistokes (cSt) that varies depending on the temperature of measurement. At 40°C, the first ester preferably has a viscosity of about 410-510 cSt more preferably about 430-490 cSt and still more preferably about 460 cSt. When measured at 100°C, the first ester preferably has a viscosity of about 35-45 cSt, more preferably about 37-43, and still more preferably about 40 cSt. The viscosity index (VI) of the first ester is preferably about 120-150, more preferably about 130-140, and still more preferably about 135.

The second ester comprises the reaction product of reactants comprising trimer acid, second polyhydric alcohol and second monohydric alcohol or reactive equivalents of one or more. This second ester has a low acid number, that is, an acid number of less than 25, preferably less than 20, 15,10,5.2, or 1. It has a viscosity, as measured in centistokes (cSt) that varies depending on the temperature of measurement. However, at any temperature below 100°C, the first ester preferably has a greater viscosity than the second ester. At 40°C, the second ester preferably has a viscosity of about 280-380 cSt, more preferably about 300-360 cSt, and still more preferably about 330 cSt.

When measured at 100°C, the second ester preferably has a viscosity of about 28-38 cSt, more preferably about 30-36, and still more preferably about 33 cSt.

The viscosity index (VI) of the second ester is preferably about 125-160, more preferably about 130-150, and still more preferably about 145

In a preferred aspect of the present invention, the first monohydric alcohol is selected from the group of C1015 primary monohydric alcohols; the second monohydric alcohol is selected from the group consisting of C8 monohydric alcohols; and the first and second polyhydric alcohols are selected from C315 polyhydric alcohols.

In one aspect, the present invention provides a blend of two esters, namely first and second esters, wherein the first ester has a greater viscosity than the second ester. In one aspect, the blend has a viscosity of 30-50 cSt at 100°C, while in another aspect the blend has a viscosity of about 40 cSt at 100°C. In a preferred embodiment, the first monohydric alcohol is selected from the group of CI-15 primary monohydric alcohols; the second monohydric alcohol is selected from the group consisting of C8 monohydric alcohols; the first and second polyhydric alcohols are selected from 3-15 polyhydric alcohols, and the viscosity of the composition is in the range of 30-50 cSt at 100°C.

The blend of two esters according to the present invention preferably has a low acid number, that is, an acid number of less than 25, preferably less than 20,15,10,5,2, or 1. The blend has a viscosity, when measured at 100°C, of 34-46 cSt, preferably about 36-44 cSt, and more preferably about 40 cSt. The blend is readily prepared from component esters by mixing the esters together in single vessel, preferably with some stirring to expeditiously achieve a homogeneous blend.

An ester of the present invention is readily prepared by combining the reactants, namely trimer acid, polyhydric alcohol and monohydric alcohol or a reactive equivalent of one or more, and heating this mixture until water is evolved. Any order of combination is suitable, and heating rate is not particularly important. The final heating temperature is suitably about 200°C to about 250°C. The progress of the reaction may be monitored by taking samples and measuring the acid number and molten viscosity of those samples.

Upon heating, water vapor will typically be evolved as the esterification reaction occurs. Preferably, the water vapor is condensed and removed from the reaction mixture as soon as it forms, thus driving the reaction to completion. A Dean-Stark trap is suitably used for this purpose. Alternatively, the water vapor is removed by application of a modest vacuum, e. g., about 20- 200 mm.

A catalyst may be used to speed up the rate of the esterification reaction, where suitable catalysts are well known in the art and include sulfuric acid, phosphoric acid and other inorganic acids, metal hydroxides and alkoxide such as tin oxide and titanium isopropoxide, and divalent metal salts such as tin or zinc salts. A preferred catalyst is a tin catalyst, e. g., FASTCAT 2001 catalyst. When a catalyst is present, it should be used in small amounts, e. g., less than about 5 weight percent of the total mass of the reaction mixture, preferably less than about 2% and more preferably less than about 1% of the total mass of the reaction mixture. Excessive amounts of catalyst increase the cost of preparing the ester, as well as often leave behind residue that may be harmful to the environment in which the ester is located, e. g., within a cable.

In preparing a preferred cable-protectant composition of the present invention, the blend of first and second esters as described above is combined with fumed silica. The cable-protectant composition provided herein preferably includes a high purity silicon dioxide which may be selected from amorphous silica, crystalline silica, fumed silica, flint, quartz, and combinations thereof. A preferred silicon dioxide is a fumed silica.

Suitable furned silica is commercially available from a number of sources, including Cabot Corporation (Cabot Corporation Boston, MA: &commat, cabot-corp com; which sells CAB-O-SILT" furned silicas). Wacker Silicons Corp (Adrian, MI: &commat., wacher, com, a division of Wacker-Chemie.) Tulco Inc.

(Ayer, MA. &commat), tulcocorp, com), and Degussa-Huls AG (Frankfurt am Main, Germany, &commat) degussahuels, de: which sellsAEROSIL fumed silica), Fumed silica is also described in U. S. Patent Nos. 6.193.944. 5.910.295, and 5.902.636. Furned silica may be prepared by the high-temperature oxidation of a halosilane such as tetrachlorosilane trichlorosilane or methyltrichlorosilane

An antioxidant is an optional component that may be combined with the ester blend and silica to prepare a cableprotectant composition of the present invention. If present, the antioxidant is used in a small but effective amount which is generally on the order of up to about 2 parts by weight based on the entire weight of the composition. Examples of suitable antioxidants include phenolic-based and phosphite-based antioxidants. Suitable antioxidants are well known in the art, where specific examples include, without limitation.IRGANOX 1010. 1RGANOX 1076. and IRGANOX 1035 antioxidants (each from Ciba Specialty Chemicals, Tarrytown, NY or Basel, Switzerland; &commat, cibasc. com) and MARK 2112, MARK 1500 and MARKTim 5014 antioxidants (each from Witco Corporation. Greenwich, CT; @ witco. com) The fumed silica, in combination with a blend of esters according to the present invention, will form a gelled mixture and/or demonstrate thixotropic properties, which is advantageous for a cable protectant composition. Methods to combine fumed silica with fluids other than the ester- containing compositions of the present invention, to form a cable protectant composition, are well known in the art, and are equally applicable to the formation of cable protectant composition using the ester-containing compositions of the present invention. See U. S. Patent Nos. 5,905,833; 5,902,849; 5,737,469; 5,285,513; 5,276,757; and 5,187,763; and European Patent EP 0 206 234 B1, and references cited therein, for descriptions of these methods.

The following examples are provided for purposes of illustration and are not limiting on the present invention. In the Examples, UNIDYMET 40 and 60 polymerized fatty acids are blends of dimer and trimer acid available from Arizona Chemical (Jacksonville, FL: &commat:arizonachemical, com).

UNtDYME 40 polymerized fatty acid is a distillation product from the polymerization of unsaturated tall oil fatty acids, in the form of a dark viscous liquid (Gardner color 12, viscosity at 210°F of 140 cSt), having a 35% contents of trimer acid (by gas chromatography) and a 65% contents of dimer acid.

UNIDYMET" 60 polymerized fatty acid is likewise a distillation product from the polymerization of unsaturated tall oil fatty acids, in the form of a dark very viscous liquid (Gardner color 14) having a 67% trimer acids content and a 33% dimer acids content. UNIDYMET" 60 polymerized fatty acid has an acid number of 192. EXXALTM 13 iso-tridecyl alcohol is from ExxonMobil Chemical Company (Houston, TX; @exxon. com/exxonchemical). Neopentyl glycol is from Eastman Chemicals (Kingston, TN), FASCAT organometallic catalysts, e. g., FASCATT 2001 stannous oxalate, are available from Atofina North America Inc. (Philadelphia, PA; @, elf-atochem. com). HFSC (High Flow Super Cell) is a diatomaceous earth filter aid. The term"AN"refers to acid number, which is synonymous with acid value"and may be measured by techniques well known in the art. See, e. g., ASTM D 465 (1982). Viscosity measurements are performed according to ASTM D 445 (1996) (Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids). Pour point is determined according to ASTM D 97 (1985) (Pour Point of Petroleum Oils).

Flash and Fire Points are measured according to ASTM D 92 (1997) (Standard Test Methods for Flash and Fire Points by Cleveland Open Cup).

EXAMPLES EXAMPLE 1 TRIMERACID, MONOALCOHOLAND 5 WT% NEOPENTYLGLYCOL A 3-liter flask was charged with UNIDYMETM 60 polymerized fatty acids (1000 g, 3.35 eq.), EXXALTM 13 iso-tridecyl alcohol (568 g. 2.87 eq.) and neopentyl glycol (5.0% based on UNIDYMEw 60 polymerized fatty acids, 50.0 grams, 0.96 eq.). The mixture was heated to 101 OC and FASCATTM 2001 catalyst (0.025% based on UNIDYMET" 60 polymerized fatty acids, 0.25 g) was added. The reaction temperature was increased to a maximum of 220°C and the acid value of the reaction mixture was determined to be 54.9. The reaction mixture was maintained at 220°C for 111/2 hours and vacuum was slowly applied to the mixture to maintain reflux when necessary. The vacuum was then lowered to 10 mm to remove any remaining volatile materials from the reaction mixture. The reaction mixture was then cooled to 130°C and filtered through HESC using Whatman &num:1 paper (under vacuum) to give a product with an acid value of 1.1, a Gardner color of 14-, a viscosity at 40°C of 516.8 cSt, a viscosity at 100°C of 44.1 cSt, a viscosity index of +138, a pour point of 33°C, a flash point of 580°F, and a fire point of 650°F.

EXAMPLE 2 TRIMERACID, MONOALCOHOLAND 3.5 WT% NEOPENTYLGLYCOL A 5-liter flask was charged with UNIDYMEm 60 polymerized fatty acids (1703 g. 5.7 eg.), EXXALTM 13 iso-tridecyl alcohol (1357 g. 6.85 eg.), and neopentyl glycol (5% based on UNIDYME 60 polymerized fatty acids, 59.6 g, 1.15 eq.). The mixture was heated to 107°C and FASCATTM 2001 catalyst (0.025% based on UNIDYMET" 60 polymerized fatty acids. 0.43 g) was added. The reaction temperature was increased to a maximum of 220°C and the acid value of the reaction mixture was determined to be 40.7. The reaction mixture was maintained at 220°C for 51°L hours and vacuum was slowly applied to the mixture to maintain reflux when necessary. The vacuum was then lowered to 1 mm to remove any remaining valuelie materials from the reaction mixture. The reaction mixture was cooled to 130°C and filtered through HFSC using Whatmen #1 paper (under vacuum) to give a product with an acid value of less than 2.0 and a viscosity at 40°C of

EXAMPLE 3 TRIMERACID. MONOALCOHOLAND 2 5 WT% NEOPENTY-LGLYCOL A 3-titer flask was charged with NIDDYMETM 60 polymerize ditty acids (1054 9, 3.5 seq.), EXXAIIII 13 13 so bridecy alloohol (598.7 g. 3.0 seq.) and neopenty diycol (2.5% based on UNIDYME 60 polymerized fatty acids, 26.4 g. 0.51 eq.). The mixture was asketed to 117°C and FASCATTM 2001 catalyst (0.6% based on UNIDYME 1m 60 polymerized fatty acids, 0.51 g) was acided The reaction temperature was increased to 220°C and the acid value of the reaction mixture was determined be 30.4 The reaction mixture was maintained at 220°C for 11 hours and vacuum was slowly applied to the mixture to maintain reflux when necessary. The vacuum was then lowered to remove any remaining volatile materials from mixture. The reaction mixture was cooled to 130°C and filtered through HFSC using Mixturam Anum; paper (under vacuum) to give a product with a viscosity at 40°C of 36.9 cSt, a viscosity at 100°C of 33.7, a viscosity index of 132. a specific cravity of 0.999 and a Gardner color of 14+.

EXAMPLE 4 BLEACHED TRIMERACID, MONOALCOHOLAND 5 WTs. NEOPENTYLGLYCOL A 3-lifer flask was charged with UNIDYMETM 60 polymerized fatty acids (600 g, 2.0 eq.) that had been previously bleached with palladium catalyst, EXXALTM iso-tridecyl alcohol (316 g, 1.6 eq.) and neopentyl glycol (5.0% based on UNIDYMETM 60 polymerized fatty acids. 30 g, 0.58 eq.). The mixture was heated to 113°C and FASCATTM 2001 catalyst (0.025% based on UNIDYMET* 60 polymerized fatty acids, 0.15 g) was added. The reaction temperature was increased to 220°C and the acid value of the reaction mixture was determined to be 38.6. The reaction mixture was maintained at 20°C for 91/2 hours at which point vacuum was applied (of 1 mm) to remove any remaining volatile materials.

The reaction mixture was cooled to 130°C and filtered through HFSC using Whatman #1 paper (under vacuum) to give a product with a viscosity at 40°C of 554 cSt, an acid value of 2.4, a Gardner color of 8-, a pour point of

EXAMPLE 5 DIMER/TRIMERACID, MONOALCOHOLAND 5% NEOPENTYLGLYCOL A 3-liter flask was charged with UNIDYMETM 40 polymerized fatty acids (1041 g, 3.53 eq.), EXXALTM iso-tridecy latchol (601 g, 3.04 eq.) and neopentyl glycol (5% based on UNIDYME 40 polymerized fatty acids, 52.1 g, 1.0 eq.). The mixture was heated to 1.3 and FASCATTM 2001 catalyst (0.025% based on UNIDYMET 40 polymerized fatty acids, 0.26 g) was added.

The reaction temperature was increased to 220°C and the acid value of the reaction mixture was determined to be 63.6. The reaction mixture was maintained at 220°C for 13 hours and vacuum was slowly applied to the mixture to maintain reflux when necessary. The vacuum was then lowered to 1 mm to remove any remaining volatile materials from the reaction mixture. The reaction mixture was cooled to 130°C and filtered through HFSC using Whatman #1 paper (under vacuum) to give a product with a viscosity at 40°C of 462°Cst, a viscosity of 136°C of 405°Cst, a viscosity of 136°C of 405°Cst, a viscosity of 136°C, a flash point of 50°C, a flash point of 50°C, a flash point of 50°C, a flash point of 50°C of 405°Cst.

EXAMPLE 6 DIMERI/TRIMERACID, MONOALCOHOLAND25% NEOPENTYLGLYCOL A 3-liter flask was charged with UNIDYMETM 40 polymerace fatty acids (1037 g. 3.52 e.g.), EXXALTM 31 is boriticely allochol (716.8 g. 3.62 eq.) and neopentyl glycol (2.5% based on UNIDYMEW 40 polymerized fatty acids, 25.9 g. 0.5 eq.). The mixture was heated to 110 CO and FASCATTM 2001 catalyst (0.05% based on UNIDYME 40 polymerized fatty acids, 0.5 g.) was added. The reaction temperature was increased to 220°C and the acid value of the reaction mixture was determined to be 421. The reaction mixture was maintained at 220°C for 11 hours and vacuum was slowly applied to the mixture to maintain reflux when necessary. The vacuum was then lowered to 2 mm to remove any remaining volatile materials from the reaction mixture. The reaction mixture was cooled to 130°C and filtered through HFSC using Whatman & num: paper (under vacuum) to give a product with a viscosity at 40°C of 368 2 cSt, a viscosity of 100°C of 53.7 cSt. a viscosity of 100°C of 53.7 cSt. a viscosity of 100°C of 53.7 cSt.

EXAMPLE 7 UNIDYME 40,2-ETHYLHEXANOLAND 5% NEOPENTYLGLYCOL A 3-liter flask was charged with UNIDYMETM 40 polymerized falty acids (886, 3 eq.), 2-ethylexand (335, 2.58 eq.), and neopenly glycov (6.6% based on UNIDYMETM 40 polymerized falty acids. 44.3 g. 0.85 eq.), The mixture was heated to 110°C and FASCATM 2001 catalyst (0.025 % based on UNIDYME 40 polymerized falty acids. 0.22 g) was added. The reaction temperature was increased to 220°C and the acid value of the reaction mixture was determined to be 63.9. The reaction mixture was maintained at 220°C for 7 hours and vacuum was slowly applied to the mixture to maintain reflux when necessary. The vacuum was then lowered to 10 mm to remove any remaining votatile materials from the reaction mixture. The reaction mixture was maintained flux with the control of 150°C for 33.7 cSt, a viscosity index of 143, a acid value of 23.8 a Gardner color of 15, a por point of 39°C, a flash point of 50°C, and as free point of

000 r

EXAMPLE 8 UNIDYME 40, 2-ETHYLHEXANOLAND 5% NEOPENTYLGLYCOL A 5-life flask was charged with UNIDYMETM 40 polymerized fitty acids (1933 9, 6.5.6.9), 2-enthylavsanol (1932, 9.5.6.9.), and neopentyl glycol (5% based on UNIDYMETM 40 polymerized fatty acids. 9.6.7.9, 1.9.9.). The mixture was heated to 110°C and F-SCATTM 2001 cataryst (0.25% based on UNIDYMETM 40 polymerized fatty acids. 0.48 g) was added. The reaction temperature was increased to 220°C and the acid value of the reaction mixture was maintained at 220°C for 6 hours and vacuum was slowly applied to the mixture to maintaine at 220°C for 6 hours and vacuum was slowly applied to the mixture to maintain reflux when necessary. The vacuum was then lowered to 1 mm to remove any remaining volatile materials from the reaction mixture. The reaction mixture was cooled to 130°C and filtered through HFSC using Whatman #1 paper (under vacuum) to give a product with a viscosity at 40°C of 321.5 cSt. an acid value of 2.1. and a Gardner color of ca.

EXAMPLE 9 UNIDYME 40,2-ETHYLHEXANOLAND 10% NEOPENTYLGLYCOL A 3-liter flask was charged with UNIDYMETM 40 polymerized falty acids (163 9,3 94 eq.),2-ethlyhexanol (265 9, 2.04 eq.) and neopentyl glycol (10% based on UNIDYMETM 40 polymerized fatty acids. 116,3 g, 2.24 eq.). The mixture was heated to 111 °C and FASCATTM 2001 catalyst (0.025% based on UNIDYME 40 polymerized fatty acids, 0.29 g) was added. The reaction represent was increased to 220°C and the acid value of the reaction mixture was determined to be 477. The reaction mixture was maintained at 220°C for 12 hours and vacuum was slowly applied to the mixture to maintain reflux when necessary. The vacuum was then lowered to 2 mm to remove any remaining volatile materials from the reaction mixture. The reaction mixture was cooled to 130°C and filtered through HFSC using Whatman #1 paper (under vacuum) to give a product with a viscosity at 40°C of 1520 cSt.

EXAMPLE 10 ESTER BLEND The ester from Example 2 (75% by weight) was combined with the ester from Example 1 (25% by weight) to give an ester blend with the following characteristics: an acid value of 1.3, a viscosity at 40°C of 40.3.6 cSt, a viscosity at 00°C of 36.3 cSt, a viscosity index of 134, a hydroxyl number of 10.2, a flash point of 555°F, a fire point of 650°F, a pour point of 33°C, a Gardner color of 13-, and an iodine value of 10.4.

EXAMPLE 11 ESTER BLENDS Three blends were prepared as identified in the Table below. The viscosity of each blend was also determined. W. & Sets of CSxmple 7 W. & Ests of Example 5 Viscosity at 40°C (Cs)l 8 0.351.7 50 40 377. 0.40 60 403.0 EXAMPLE 12 ESTER BLEND The ester from Example 8 (40% by weight) and the ester from Example 8 (40% by weight) where combined to give a final product with a viscosity at 40°C of 399 CSI, a viscosity of 138, an acid value of 1.3, a Gardner color of 13, a flash point of 50°F, a fire point of 670°F and a pour point of 33°C.

EXAMPLE 13 A blend was prepared from the ester of Example 8 (60% by weight) and the ester of Example 5 (20% by weight). The final product had a viscosity at 40°C of 349 cSt, a viscosity at 100°C of 34.5 cSt, a viscosity index of 142. an acid value of 17. a Gardren color of 13, a flash point of 580°F, a fire point of 675°F, and a pour point of 530°F.

EXAMPLE 14 ESTER.POLYPROPYLENECOMPATIBILITY The compatibility of ester blends of the present invention with polypropylene was determined by measuring the extent to which ester absorbed into polypropropoylene when the two materials were maintained in contact for several weeks. More specifically, samples of polypropylene extruded strips having dimensions 0.7 mm thickness and 20 mm width were weighed (each strip weight approximately 3 grams). A known weight of esters, in the range of 30 grams, was added to an aluminum pan of dimensions 63.5 mm x 17.5 mm. An individual strip of polypropylene was placed in the aluminum pan with the esters, and the pan was covered with aluminum foil.

The sample was maintained at 80°C for a predetermined period. First Ester Second Ester % Wt Gain % Wt Gain % wt Gain Wt Vt Wt Gain % wt Gain Wt Wt Gain % wt Gain Wt Gain % wt Gain %

All publications and patent applications mentioned in this specification are herein incorporated by reference to the same extent as if each individual publication or patent application was specifically and individually incorporated by reference.

From the foregoing it will be appreciated that, although specific embodiments of the invention have been described herein for purposes of illustration, various modifications may be made without deviating from the spirit and scope of the invention. Accordingly, the invention is not limited except as by the appended claims.



IP SERVICES



nome If Services FatentScope Patent Search



Search result: 1 of 1

(WO/2002/088208) POLYBASIC ACID ESTERS AND THEIR USE IN CABLES

Biblio, Data Description Claims National Phase Notices Documents

CLAIMS 1. A composition comprising a blend of first and second esters, the first ester comprising a reaction product of first reactants comprising trimer acid or reactive equivalent thereof, first polyhydric alcohol or reactive equivalent thereof, and first monohydric alcohol or reactive equivalent thereof, the first monohydric alcohol being selected from the group of C8-18 monohydric alcohols or reactive equivalents thereof, the second ester comprising a reaction product of second reactants comprising trimer acid or reactive equivalent thereof, second polyhydric alcohol or reactive equivalent thereof, and second monohydric alcohol sor reactive equivalent thereof, the second monohydric alcohol selected from the group of C810 monohydric alcohols or reactive equivalents thereof; the first and second monohydric alcohols being non-identical.

- 2. The composition of claim 1 wherein the first reactants and the second reactants each further comprise dimer acid.
- 3. The composition of claim 2 wherein the first reactants and the second reactants each comprise dimer acid and trimer acid, in a dimer acid: trimer acid weight ratio of 20: 80 to 80: 20, the weight ratio independently selected in each of the first and second reactants.
- 4. The composition of any of claims 1-3 wherein the trimer acid is hydrogenated trimer acid.
- The composition of any of claims 1-4 wherein the first monohydric alcohol is selected from C1014 primary monohydric alcohols.
- 6. The composition of any of claims 1-5 wherein the first monohydric alcohol is iso-tridecyl alcohol
- 7. The composition of any of claims 1-6 wherein the first and second polyhydric alcohols are each selected from C3-15 polyhydric alcohols.
- The composition of any of claims 1-6 wherein the first and second polyhydric alcohols are each independently selected from pentaerythritol, di-pentaerythritol, tin-pentaerythritol, trimethylolpropane, ethylene glycol and neopentylgylol.
- 9. The composition of any of claims 1-6 wherein the first and second polyhydric alcohols are each neopentyl glycol.
- The composition of any of claims 1-6 wherein the second monohydric alcohol is selected from 2-ethylhexanol, 2octanol and cyclohexyl alcohol.
- 11. The composition of any of claims 1-6 wherein the second monohydric alcohol is 2-ethylhexanol.
- 12. The composition of any of claims 1-11 wherein the first ester has a greater viscosity than the second ester.
- 13. The composition of any of claims 1-12 having a viscosity of 30-50 cSt at 100°C.
- 14. The composition of any of claims 1-13 having a viscosity of about 40 cSt at 100°C.
- 15. The composition of claim 1 wherein the first monohydric alcohol is selected from the group of C1015 primary monohydric alcohols; the second monohydric alcohol is selected from the group consisting of C8 monohydric alcohols; the first and second polyhydric alcohols are selected from C3-15 polyhydric alcohols, and the viscosity of the composition is in the range of 30-50 c5t at 100°C.

- 16. A method for preparing an ester composition having a viscosity in the range of 30-50 cSt at 100°C, the method comprising the steps of a) preparing a first sett, the first setor comprising a reaction product of first reactants comprising time reaction product of first reactants comprising timer acid, first polyhydric alcohol and first monohydric alcohol; the first monohydric alcohol selected from the group of Cg-18 monohydric alcohols; the preparing a second ester, the second ester comprising a reaction product of second reactants comprising timer acid second second second reactants comprising timer acid second seco
- 17. The method of claim 16 wherein the first reactants and the second reactants each further comprise dimer acid, in a dimer acid, trimer acid weight ratio of 20:80 to 80:20, the weight ratio independently selected in each of the first and second reactants.
- 18 The method of any of claims 16-17 wherein the trimer acid is hydrogenated trimer acid.
- 19. The method of any of claims 16-18 wherein the first monohydric alcohol is selected from C10-14 primary monohydric alcohols.
- 20. The method of any of claims 16-19 wherein the first and second polyhydric alcohols are each selected from C315 polyhydric alcohols.
- 21, The method of any of claims 16-20 wherein the second monohydric alcohol is selected from 2-ethylhexanol, 2-octanol and cyclohexyl alcohol.
- 22. The method of any of claims 16-21 wherein the first ester has a greater viscosity than the second ester.
- 23. The method of claim 16 wherein the first monohydric alcohol is selected from the group of C10-15 primary monohydric alcohols: the second monohydric alcohol is selected from the group consisting of C8 monohydric alcohols; the first and second polyhydric alcohols are selected from C3-15 polyhydric alcohols; and the first ester has a greater viscosity than the second ester.
- 24. A composition prepared by the method of any of claims 16-23.
- 25. A cable-filling composition comprising silica and a blend of first and second esters, the first ester comprising a reaction product of first reactants comprising interner acid, first polyhydric alcobol and first monohydric alcohol; the first monohydric alcohol selected from the group of C8-18 monohydric alcohols; the second ester comprising a reaction product of second reactants comprising timer acid, second polyhydric alcohol and second monohydric alcohol; the second monohydric alcohol selected from the group of C610 monohydric alcohols; the first and second monohydric alcohol selected.
- 26. The composition of claim 25 wherein the first monohydric alcohol is selected from the group of C10-15 primary monohydric alcohols: the second monohydric alcohol is selected from the group consisting of C8 monohydric alcohols; the first and second polyhydric alcohols are selected from C3-15 polyhydric alcohols, and the viscosity of the blend is in the range of 30-50 cSt at 100°C.
- 27. A method for insulating the contents of a cable, the method comprising a) preparing a cable-filling composition comprising silica and a blend of first and second esters; the first ester comprising a reaction product of first reactants comprising timer acid. First polyhydric alcohol and first monohydric alcohol; the first monohydric alcohol selected from the group of C8-18 monohydric alcohols; the second ester comprising a reaction product of second reactants comprising timer acid, second polyhydric alcohol and second monohydric alcohol; the second monohydric alcohol selected from the group of C6-10 monohydric alcohols; the first and second monohydric alcohols being non-identical; b) placing the cable-filling composition of step a) into a filter opticable.
- 28. The method of claim 27 wherein the cable is a fiber optic cable.
- 29. A cable comprising a cable-filling composition: the cable-filling composition comprising silica and a blend of first and second eaters; the first seter comprising a reaction product of first reactants comprising trimer acid, first pohyhydric alcohol and first monohydric alcohol : the first monohydric alcohol selected from the group of C8-18 monohydric alcohols: whe second seat comprising a reaction product of second reactants comprising timer acid. second polyhydric alcohol and second monohydric alcohol : wherein the first and second monohydric alcohols: wherein the first and second monohydric alcohols: wherein the first and second monohydric alcohols : wherein the first and second monohydric alcohols : wherein the first and second monohydric alcohols : wherein the first and second monohydric alcohols are non-identical.

30. The cable of claim 29 wherein the cable is a fiber optic cable.

Exhibit D

A copy of the Federal Express tracking confirmation for the April 27, 2007 letter from J.M. Gilbreth to Nancy Mosby.

FedEx | Track Page 1 of 2



Select format: - HTML
Add personal message:

English Text

Wireless

FedEx | Track Page 2 of 2

Not available for Wireless or non-English characters.

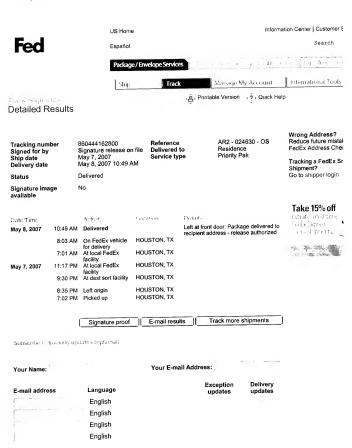
By selecting this check box and the Submit button, I agree to these ${\tt Termis}$ and ${\tt Conditions}$

Global Home | Service Info | About FedEx | Investor Relations | Careers | fedex.com Terms of Use | Privacy Policy
This site is protected by copyright and trademark laws under US and International law. All rights reserved. © 1995-2007 FedEx

Exhibit E

A copy of the Federal Express tracking confirmation for the May 7, 2007 letter from J.M. Gilbreth to Nancy Mosby.

FedEx | Track Page 1 of 2



Wireless

Text

Select format:
¬ HTML

Add personal message:

FedEx | Track Page 2 of 2

Not available for Wireless or non-English characters.

By selecting this check box and the Submit button, I agree to these Terms and Conditions

Global Home | Service Info | About FedEx | Investor Relations | Careers | fedex.com Terms of Use | Privacy Policy
This site is protected by copyright and trademark laws under US and International law. All rights reserved.

Exhibit F

A copy of the May 8, 2007 letter from Nancy Mosby to J.M. Gilbreth.

May 6, 2007

Mr. J. M. (Mark) Gilbreth Frost Bank Building 6750 West Loop South Suite 920 Bellatre, Tonge 77101

RE: ARZ - 024630-N1 (Application No. 11/637,206, PCT/US02/12961) Polybasic Acid Esters and Their Use in Cables

Dear Sir:

Thank you for your letter dated April 27, 2007. In response please see the enclosed letter dated April 23, 2002 that expresses my continued wishes to have nothing to do with the above referenced patent, Arizona Chemical, or anyone associated with Arizona Chemical. Please note that the original was mailed directly to my former supervisor Lloyd Nelson who was employed by Arizona Chemical at the time.

To me, the previous letter is crystal clear. I do not understand why this matter was not resolved years ago and consider it to be so now. Any further contact or attempt to contact me by you or anyone associated with Arizona Chemical or their designees will be looked upon as harassment and will be handled as such.

Thank you,

Malley

April 23, 2002

P.O. Box 2553 Savannah, Georgia 31402

To Whom it May Concern:

I, Nancy D. Mosby, hereby withdraw my name from everything connected with a patent being applied for by International Paper concerning an ISO VG 400 product (trimerate) developed by Lloyd Nelson and myself. Although one of my contributions (blending two different products (that I also helped develop) to achieve the desired viscosity of 400 cSt at 40°C) led directly to the development of the product being patented, I do not desire any credit or recognition in relation to this patent, nor do I desire to retain any potential benefits now or in the future associated with this patent.

Thank you for complying with my request. If you must contact me for anything further, you can direct a letter to the address listed above. I expect nothing less than common courtesy from you, and request nothing more.

Sincerely,

Nancy D. Mosby



SO WAY SOOT ON 2 L

Mr. J. M. (Mark) Gilbreth Frost Bank Building 6750 West Loop South Suite 920 Bellaire, Texas 77401

SO4 FO4

Hardinglad than Hadan hapter data tatal

77401+4117